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DEVELOPMENT OF A COLD CORRUGATING PROCESS
FINAL REPORT

SECTIONS I & II

By
Clyde H. Sprague

May, 1985

Work Performed Under Contract No. DE-AC02-79CS40211

The Institute of Paper Chemistry
Appleton, Wisconsin

TECHNICAL INFORMATION CENTER
U. S. DEPARTMENT OF ENERGY

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The Institute of Paper Chemistry
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Office of Industrial Programs CE-12
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Conservation and Renewable Energy

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EXECUTIVE SUMMARY

In the early 1970's energy was scarce and energy prices were rising rapidly. Some boxplants were shut down for periods of time because of energy shortages. Many expected these conditions to worsen with time. Work on a cold corrugating process, described in this report, was initiated in the mid-1970's, partially in response to the adverse energy situation. As conceived at the time, cold corrugating offered a 90% reduction in process heat demands and significant advantages in electrical drive energy, productivity and waste reduction.

Corrugating involves two process elements: fluting of the corrugating medium and bonding of the medium to the liners. Two very different bonding processes are required, one at the single facer, the other at the double backer. For cold corrugating, each of these process elements must be accomplished with little or no process heat. Two earlier projects served as precursors for cold corrugating. In one of these projects, various lubricating agents, abraded onto the medium in solid form, were used successfully as adjuncts to or replacements for preconditioning in the hot fluting process. Extending this concept to enable successful cold forming was believed possible.

In another project, thermochemically converted starch adhesives were investigated for use on "warm" corrugators, operated with hot corrugating rolls, but with a cold pressure roll. Although the result of limited development, this adhesive worked reasonably well in single facing trials. This adhesive, modified for cold operation, and a lubricating agent to ease cold forming of the medium, constitute the essential ingredients of cold corrugating. These basic elements were developed and evaluated in a series of projects and trials in the

laboratory, on a pilot machine and, finally, on a commercial prototype machine. This work is summarized below and described in detail in the body of this report.

1. COLD FLUTING

Development of a cold fluting process, started in 1973, was based on the results from earlier investigations of the use of solid lubricants for hot corrugating. Numerous solid agents suitable for use under room temperature conditions were evaluated for their ability to reduce friction and enhance cold fluting performance. From this screening work blends of materials were selected for further study. The following blend was finally selected for use on the basis of its excellent performance in cold forming trials.

93% low melting point paraffin wax
5% powdered graphite
1% silicone oil
1% stearin

The principal function of the medium treatment agent (MTA) is to reduce the friction coefficient between the medium and the corrugating rolls to prevent tension build up and consequent fracture or damage of the medium. Stearin is included to improve release of the medium from the corrugating rolls and thereby to lessen picking, silicone oil is included because it is beneficial in reducing the high-lows produced during fluting, and graphite is included to reduce friction.

For application, this agent is molded into solid bars and abraded onto both sides of the medium web. An excellent applicator system for this purpose is described in the report. Only a very small amount of the agent is required for effective functioning - usually less than can be applied successfully. Hence, the cost of lubricating agent applied tends to be determined by the limitations of the application process and not by the requirements of cold forming. Despite this limitation, the cost is negligible when proper attention is paid to the applicator system.

Through use of this or other suitable MTA's, cold fluting of most 26#/MSF mediums can be accomplished without fracture up to at least 650 fpm. Some limited trials have pushed the top speed to 1000 fpm, but most test work was intentionally limited to speeds of 650 fpm or less. Only a few trials were conducted with heavier basis weight mediums. These are more difficult to form without fracture, but several were successfully cold formed at 650 fpm.

When compared to hot forming, cold forming consistently produces the following single face structural performance.

Property	Hot	Cold
Caliper, inch	0.154	0.156
Edgewise compression, lb/inch	30	30
Flat crush, lb/inch^2	33	29

These comparisons are based, in part, on an exhaustive evaluation of about 35 different corrugating mediums tested under hot and cold corrugating conditions on the same machines. However, similar and consistent data were obtained in both the pilot and commercial prototype machines. Cold corrugating compares favorably in all areas except flat crush. However, when viewed individually, some mediums exhibit comparable performance under both processes, thus suggesting papermaking approaches to improving flat crush under cold fluting conditions. For cold fluting, flat crush does not correlate well with Concora values.

As suggested by the efficacy of MTA's in promoting cold forming, the medium friction coefficient is a critical factor. In fact, it is the only medium property that correlates strongly with cold fluting performance. In the broad test samples, all mediums having a friction coefficient less than 0.4 were successfully cold formed without use of medium treatment agents; all with coefficients greater than 0.5 required pretreatment. Mediums between these limits could not be distinguished on the basis of the friction coefficent. These results further illustrate the opportunity to adapt medium properties for cold fluting performance.

A fundamental study of forming, initially intended to provide an understanding of the differences between hot and cold fluting performance. showed that both processes severely degrade the initial strength potential of the medium. Losses range up to 40% in the machine direction (MD), and up to 20% in the cross direction (CD) in both processes. This discovery prompted redirection of the investigation to seek an understanding of the degradation mechanism rather than the differences between the two fluting processes. Damage is caused primarily by bending strains induced during forming and aggravated by tensional strains. Out-of-plane shear strains in the medium tend to reduce the severity of damage. Medium properties can be altered during manufacture to both improve base strength and reduce strength losses due to fluting. Laboratory test sheets made from a representative medium furnish according to the requirements for good forming show 20% higher base strength and 20% better strength retention than commercial mediums. The net result is a 40% improvement in end-use performance.

This result is now finding its way into practice resulting in improved performance in hot corrugating. Ultimately as the market place allows, this benefit may be taken, at least partially, as a reduction in fiber amount or quality. It is possible that the resulting energy savings will far exceed those originally projected for cold corrugating.

2. BONDING

All of the early work on cold-setting adhesives was based on thermochemical conversion of pearl corn starch. Such adhesives thicken or set back, partially irreversibly, upon cooling and, hence, must be held and applied at elevated temperatures to maintain a workable viscosity. Bond strength develops partly by cooling, but mostly by water loss. For lack of equipment, only single face bonding was studied until midway through the project. At the single facer, green bond development is driven by mechanically induced dewatering of the adhesive at the pressure roll nip (bonding nip). Setback adhesives prepared at 20-25% solids levels produced generally acceptable green and final bond strengths when used on a "warm" corrugator. Some bond brittleness was encountered, however. Limited tests with higher solids adhesives were also conducted, but with somewhat mixed results.

For fully cold single facing (no heat or steam) the pearl starch setback adhesive was modified on the basis of many experiments to about 33% solids. All conversion chemicals except ammonium persulfate (AP) were deleted from the formula, and the cooked adhesive was adjusted to about pH 9 with NaOH. With this type of adhesive, the laboratory single facer was routinely operated up to 600 fpm and, occasionally, at much higher speeds. Green bonds and pin adhesion levels were satisfactory; bond failure in the medium was common with liner failure occurring only occasionally. These results were regarded as encouraging, although application rates were not accurately known and, as later data showed, were excessive.

3. PILOT TRIALS

Based on the encouraging results from the laboratory work, pilot scale trials on a commercial scale machine were undertaken. The objectives of this phase were to evaluate the double face bonding process and to identify the problems of commercial application of cold corrugation. A very simple conversion of a commercial hot machine provided the test bed for this work. Early tests with the full-scale single facer provided results equivalent to those obtained in the laboratory, but, again, equipment limitations precluded operation at low rates of adhesive application. As was shown later, the excessive application rates in both the laboratory and pilot equipment masked a bond brittleness problem.

Initial trials on the pilot machine provided the first opportunity to evaluate the double face (DF) bonding process. These trials were carried out using the same adhesive formula used at the single facer at 33% solids. Excessive and uncontrolled adhesive application rates confounded interpretation of the results from the first trial. Despite this, it was immediately evident that the cold corrugating adhesive was slow to set up under double backer conditions, leading to slitter-edge delamination at speeds above 300-400 fpm. During most of the pilot trials period, attention was focused on proper adhesive application at the glue machine and on improving the double facer bonding system to improve speed.

Because the pilot system was poorly suited to detailed, quantitative study of the DF bonding system, a double backer simulator was developed. This simulator provided an effective simulation of all aspects of the DF bonding system, including provisions for measuring the effect of bond age (time) on bond strength. The simulator was used extensively and effectively to study the DF bonding issue. Five mechanisms for increasing the rate of development of the DF bond were identified in these tests:

1. Increase adhesive solids content to the maximum possible level, about 39% for jet-cooked, setback adhesives based on pearl starch.
2. Use the maximum possible adhesive application temperature, around 190-195°F.
3. Use the minimum possible adhesive application rate, constrained by final bond strength requirements.
4. Use the maximum combining pressure, constrained by board crushing and caliper loss.
5. Preheat the liner and/or the single face web. This has a large, positive impact on machine operating speed for a small increase in energy cost.

Because of the need to retain full hot production capacity in the pilot machine, only very simple, limited conversions were possible. As a consequence, items 4 and 5 were only partially achieved, and then only through use of extraordinary experimental procedures. As a result, consistent production exhibiting the full potential of cold corrugating was never achieved on the pilot machine. It was believed, however, that these deficiencies could be removed in a commercial prototype system.

A few of the late trials included, in a limited fashion, all of the factors believed to improve DF bonding. From these, some board of near-marketable quality was produced at the project target speed of 600-650 fpm. Representative performance data, referenced to typical hot production, are shown below.

Property	Cold	Hot
Caliper, inch	0.164	0.161
Single face bond, psi	16	17
Double face bond, psi	16	14
Edgewise compression, lb/inch	49.0	50.0
Flat crush, lb/inch^2	30.0	35.0

With the exception of flat crush, as discussed before, these data show comparable performance for the cold and hot processes. Although not revealed in these test data, the double face bonds exhibited some slippage (relative motion between the flute tip and liner after initial contact was made). Slippage reduces bond strength, bond strength per unit of adhesive (effectiveness), and bond toughness. Despite the slippage-induced degradation the DF bonds were strong and produced some fiber tear on failure. Although the cause of the slippage was incorrectly diagnosed at the time, the commercial prototype double backer design was expected to eliminate it (and probably would have, had it been used). All of the simulator results were confirmed in the pilot trials.

The pilot trials were intended to identify - but, not necessarily solve - the problems of commercial application of the cold corrugating process. From the pilot trials' results and experiences, the specifications for commercial prototype equipment were prepared. Experience with the prototype system showed that those specifications were largely correct, thus verifying the efficacy of the pilot trials in fulfilling the objective originally set for them. The lone exception was the single face bond toughness issue, not fully revealed in the pilot trials.

Early plans for the development of the cold corrugating process included a true pilot machine at The Institute of Paper Chemistry. Funding for the pilot system was denied by the industry, however, and the Institute sought the joint support of the Department of Energy and the industry to pursue the commercial prototype approach as an alternative. This phase of the project was initiated in mid-1979, about 18 months before the conclusion of the pilot scale trials.

4. COMMERCIAL PROTOTYPE PHASE

The objective of this phase of the project was to demonstrate the commercial viability of the cold corrugating process by providing and evaluating the first equipment designed specifically for cold corrugating. For this purpose, the Union Camp Corporation was subcontracted to provide its Savannah box plant as the host site. An existing 3 single facer machine was modified to allow cold single wall production. Four major items of equipment were supplied: an adhesive makeup and delivery system, a single facer, a glue machine, and a double backer. An extensive data acquisition and sensor system was also included. For all of the early equipment installation and checkout steps, combined board trials made use of the existing double backer fitted for cold running. This plan was designed to avoid losses in hot production until the cold system could take over at least limited commercial production. This point was never reached and the new, cold double backer was never installed.

For acquisition of the equipment, detailed specifications were prepared on the basis of the laboratory and pilot plant data and experiences. Vendors were selected on the basis of detailed proposals and price. The equipment was purchased for delivery and installation on a turnkey basis, although some modification was expected because of the prototype nature of the equipment. However, most items of equipment required extensive modification, some of it on conventional (not significantly impacted by cold corrugating) items. These problems proved very costly and time consuming, often requiring the development of special techniques for proper diagnosis. As a consequence, most of the trials actually conducted were aimed at troubleshooting and not at overall evaluation of the cold corrugating system.

In the adhesive system many of the original details of sequencing and control had to be changed. These problems proved relatively easy to correct, but difficult to diagnose. At the single facer, serious problems were encountered with roll stack crowning, with tension control, and with all aspects of the adhesive applicator system. The latter system was eventually replaced in its entirety after several intermediate rebuilds failed to yield adequate results. Control of adhesive application rates and uniformity, severe contamination, vapor leakage and generally unsatisfactory mechanical design and operation were all problem areas. At the glue machine, the original adhesive metering scheme proved unworkable and was abandoned in favor of a single pattern gravure roll and trailing wiper blade. Numerous problems were encountered with the existing double backer, mostly related to the crude implementation of drag-reducing schemes, preheating and top-belt loading. These problems were aggravated by the poor mechanical condition of the base machine.

Late in 1982 and early in 1983 a short series of combined board trials was conducted. Although known deficiencies remained in the system, the project team deemed it advisable to run these trials to more completely evaluate the process and system, and to discover any other remaining but undetected problems. The intent was to run at as near commercial conditions as possible: to learn to operate the modified equipment using the new process, to train operating crews, and to fill orders, if possible. Because of mechanical limitations in the double backer the top speed was limited to about 500 fpm, regardless of process performance.

A summary of performance results from the best of the board produced in these trials is given below.

Property	
Caliper, inch	0.162
Single face bond, psi	10.2
Double face bond, psi	10.2
Edgewise compression, lb/inch	41
Flat crush, lb/inch^2	26.8
Flexural stiffness, lb-inch	
Dx	138
Dy	58

These test data show the board to be of a generally acceptable commercial quality. However, these results were not reproduced on a consistent, day-to-day basis, partly because of deficiencies in the equipment, the latter being mostly in the crudely modified double backer. In fairness, however, it should be noted that the cold process seems to allow less latitude in equipment design and operation than the hot process.

At the conclusion of this series of trials, three issues were identified as critical to the commercial operation of the process and continuation of the project. These were roll stack crowning, adhesive application and bond toughness, all at the single facer. Installation of the new double backer was expected to resolve most of the other issues.

Roll stack crowning is not a fundamentally difficult problem nor is it complicated by cold corrugating. The industry simply lacks tools for adequate analysis and design, relying instead on trial and error methods. These methods were applied with good effect on the cold single facer, although one or two more iterations may have been necessary for fully satisfactory performance on full width (96-inch) webs.

A completely new adhesive applicator system, with a new and novel wetted width control system, was installed to address four issues: severe adhesive contamination of the corrugating rolls; uniform, consistent adhesive application; controlled (variable) adhesive application rates; and generally improved mechanical design and operation. This system was installed very late in the project, but worked extremely well to satisfy all of the objectives set for it. The wetted width control system is regarded as an attractive alternative to dams in conventional corrugating.

Despite a concerted effort, described in this report, single face bond toughness proved very elusive and was never satisfactorily achieved. Adhesive ejection from the bond zone by the pressure impulse at the bonding nip is the apparent cause of brittleness. Two approaches, both outside the scope of this project, are possible: modification of the adhesive (in an unknown way) to limit mobility at the pressure roll nip or extension of this nip to allow longer residence times and lower pressures (lower ejection forces). The latter was shown to be very effective under special laboratory conditions, but these cannot be duplicated in equipment of approximately present commercial design. Bond toughness finally emerged as the fatal flaw in the cold corrugating system, resulting in termination of the project by mutual consent of all parties involved.

Many of the aspects of cold corrugating were successfully demonstrated during the course of the project. Most of the remaining issues are believed solvable with available equipment, principally the new but unused double backer. All of the available data support the design features built into that machine; none calls for features not included. Successful commercial cold corrugating will require a resolution of the single face bond brittleness issue and, possibly, some additional attention to the contamination question. Because setback adhesives adhere to cold metal, contamination by the occasional stray droplet of adhesive is a potential problem, even in the cleanest of systems.

Although the final goal of commercial demonstration of the cold corrugating system was not realized, there are many very useful results from the project. These are fully described in the following section of this report.

As finally operated, the cold process offers about an 85% reduction in process heat compared to the 95% originally projected. Projected electrical drive energy reductions of 45% were never proven because the double backer was never installed, but substantial saving is still expected. Adhesive costs should be comparable to those for hot corrugating. The projected 2% medium savings remains valid. Long-term commercial production is needed to fully assess the warp and waste issues, but late results were encouraging. Large capital cost differentials are not expected.

With this report the long-standing efforts to develop a cold corrugating process come to a close.

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SECTION I - INTRODUCTION

A. PREFACE

This is the final report on a long-standing project to develop a cold corrugating process. It is a comprehensive report, written to document the results from this project and to satisfy the requirements of Contract DE-AC02-79CS40211 between The Institute of Paper Chemistry and the U.S. Department of Energy.

In the early 1970's, two projects were carried out which ultimately served as precursors to this cold corrugating program. In the first, special lubricants were identified as aids to hot fluting. In the second, a thermochemically converted, starch based adhesive was developed for "warm" corrugating with hot corrugating rolls and a cold pressure roll. Both projects were sponsored by the Fourdrinier Kraft Board Group (FKBG) of the American Paper Institute (API).

In 1973, FKBG sponsored a project to assess the technical feasibility of a totally cold corrugating process, based on combining and extending the forming and bonding concepts developed in the earlier projects. This was followed in 1974 with an FKBG sponsored project to further develop the setback adhesive for cold corrugating.

Based on the encouraging results from these projects, a more comprehensive project was initiated in 1976. Objectives for this project included additional fundamental process development and limited pilot trials on a simply converted commercial machine to identify the problems of commercial implementation of the process. The fundamental work in this project was sponsored as part of the funded research program of The Institute of Paper Chemistry; the pilot trials portion was supported by the Fourdrinier Kraft Board Group.

Included in this comprehensive project phase were plans for additional work on a pilot-scale machine specifically designed for cold corrugating. Midway through the pilot trials, this plan was rejected in favor of the development of a true commercial prototype system. Support for this effort was sought and received from the U.S. Department of Energy. Costs were shared by the DOE, the IPC, and the FKBG.

The objectives of this final project phase were to develop and evaluate, in a commercial context, a commercial prototype cold corrugating system and to complete the pilot trials and process development work. Under a subcontract with The Institute of Paper Chemistry, the Union Camp Corporation provided its Savannah box plant as a host site for the commercial prototype unit.

For completeness, this report covers all project phases. It is organized in four sections. An additional part of this section presents a summary of the useful developments from the project. In the second section, the two basic process elements, forming and bonding, are covered in two separate parts. Development of the forming and bonding aspects of the cold corrugating process continued throughout the duration of the project, often driven by results from pilot system or commercial prototype trials. These efforts are all described in Section II. The pilot trials work is covered in Section III.

Section IV is devoted to commercial prototype system development and evaluation and is divided into three parts. Part I covers the specification and acquisition of the system components. Part II describes the checkout tests and the modifications made to the system. Part III describes the results obtained from the system.

B. RESULTS SUMMARY

Although the project was not fully successful in demonstrating the commercial viability of the cold corrugating process, there were many very important results from the project. Many of these are directly applicable to hot corrugating. All are discussed in the body of the report. To highlight these positive developments and for convenience of reference, they are summarized in this section. Each summary is referenced to the more detailed discussion in the body of the report.

1. FUNDAMENTALS OF FLUTING

When compared with flutes formed by the hot process, cold formed flutes show higher caliper, comparable high-lows, equal or higher edgewise compressive strength and lower flat crush. For some mediums, the flat crush values are nearly equal for the two processes, for others the differences may be significant. To develop an understanding of the cause of these differences, a fundamental study of the fluting process was undertaken. Surprisingly, early results from this work showed severe degradation of medium properties in both processes. Machine direction strength (flat crush) potential was degraded by about 40%; cross machine direction strength (edgewise compressive strength) was degraded by about 20%. These rather severe strength losses were regarded as far more significant than the 12% differences in hot/cold flat crush values, and the study was redirected to developing an understanding of the strength loss mechanism.

Medium damage, the cause of the strength loss, stems primarily from bending strains induced during the fluting process. These losses are heightened by excess medium tension and reduced by shear straining of the medium. Important medium properties include MD tensile stiffness and stretch, out-of-plane shear stiffness, caliper, and the coefficient of friction. These properties must be balanced to give high initial strength and high retention of strength through the fluting process.

A full discussion of this subject is given in Section II, Part I, pages 2-99. The following paragraph describes an experimental medium designed for improved end-use performance.

2. IMPROVED MEDIUM PROPERTIES

To test the basic hypotheses developed in the fluting study, several experimental mediums were produced for evaluation. Laboratory sheets were produced on the Formette Dynamique sheet former and then corrugated on the laboratory corrugator. All of the sheets were made from representative medium furnishes and pressed and dryed to produce higher densities and higher stretch values than commercial mediums. Several commercial mediums were corrugated simultaneously for comparison.

In these tests, the experimental mediums showed about 20% higher initial strength and 20% higher retention of strength for an overall gain of 40% in end-use performance (flat crush). The processing steps used to make these improved mediums are now being applied in the manufacture of a growing fraction of the commercial medium used in hot corrugating.

These experiments are described in more detail in Section II, Part I, pages 100 to 114.

3. MEDIUM TREATMENT AGENTS FOR FLUTING

In hot fluting, the medium is preheated and steamed to temporarily raise sheet temperature and moisture. These changes cause corresponding changes in the mechanical properties of the sheet to allow fluting to take place without fracture or excess damage. Reductions in the medium friction coefficient with increases in temperature may be a principal mechanism in this process. In cold fluting, heat and steam are not available.

Of the mediums tested during this project (more than 40) about half could be formed cold (up to at least 600 fpm at reasonable tension levels without fracture) without any form of pretreatment. The remaining half required pretreatment to avoid fracture. These mediums were separated solely by friction coefficient; no other medium properties correlated well with cold runnability.

Early in the project, a medium pretreatment agent blend was developed for cold corrugating. It consists of 93% low melting point wax, 5% powdered graphite, 1% silicone oil, and 1% stearin. When abraded onto the sheet from solid bars, this agent reduces the friction coefficient and improves release from the corrugating rolls. No other, better agent has been found. The cost of the material is low; the positive effect on cold forming is dramatic.

Similar agents for hot corrugating have been developed in other projects. These have been used occasionally, sometimes with excellent results. They represent a possible alternative to conventional preconditioning, which has a small, positive effect on hot fluting.

4. MEDIUM AND SINGLE FACE DATA

As a part of this project, about 35 mediums, all 26 lb/MSF, were tested in both the hot and cold single facing processes. Exhaustive tests on the medium and the hot and cold single face samples produced large quantities of very valuable data. This data set, presented in Section II, Part I, pages 114-124, is an excellent source of information on representative properties and property variability.

5. SINGLE FACE BONDING PROCESS

Adhesive distribution around the final bond zone is controlled by an ejection process that takes place at the pressure roll nip, and not by the initial distribution of the adhesive placed on the flute tip by the glue roll. Ejection removes much of the adhesive from the bonded area leading to low bond strength, low effectiveness (low bond strength per unit of adhesive), and bond brittleness. Micrographs show that this process is important in hot corrugating. In cold corrugating, it is the dominant negative factor in single face bonding, effectively preventing the development of adequate bond toughness with present equipment and reasonable amounts of adhesive. Although the result is negative, the understanding is positive. It is a key to developing a successful cold single face bonding system, and it may be important to hot single facing as well. More details are given in Section II, Part II, pages 266-286.

In cold corrugating, green single face bonds form primarily by water loss from the adhesive; cooling induced thickening is much less important. Mechanical dewatering of the adhesive by the pressure impulse at the bonding nip is the primary mechanism. This same mechanism promotes the undesirable ejection process described above. Balancing these opposing effects is necessary for good single facer performance.

Similar arguments appear valid for hot corrugating. Gelatinization of the raw starch in the pressure roll nip is unlikely because of the very short time available for heat transfer. Furthermore, the strength of a gelled starch is inadequate to form a suitable green bond. Mechanical dewatering of the starch, especially the carrier portion, may contribute most of the green bond strength. This mechanism is very much like that in cold corrugating. Apparently, hot corrugating adhesives are less mobile and, hence, less susceptible to ejection than the cold corrugating adhesives, thus explaining the differences in final bond performance.

6. DOUBLE BACKER SIMULATOR

Typical double backing equipment is totally unsuited for the study of the double face bonding system. Such equipment does not provide for control of the bonding process or for assessment of the intermediate status of bond development. Only the final bond can be examined. For these reasons the double backer simulator was developed as a versatile tool for studying all aspects of the double face bonding system.

The simulator is a miniature double backer that simulates all of the steps in double facing including adhesive application, open time, combining, and bond strength measurement with age. All of the process conditions can be accurately controlled over wide ranges. This device has proved to be an extremely productive, useful tool for studying this bonding process. Results from it have been confirmed in actual machine trials. In addition, it provides a standard of performance as a target for real double backers. For example, simulator bonds show effectiveness levels of over 3,000,000 pounds of bond strength/pound of adhesive. Real double backers are about 40-50% as effective.

For details on the simulator, see Section II, Part II, pages 213-221.

Although designed for use with cold-setting adhesives, modest changes in the simulator should make it suitable for use with conventional corrugating adhesives as well. It has already proven valuable in laminating studies.

7. DOUBLE FACE BONDING

For aqueous-based, cold-setting adhesives, the double face bond development rate and, hence, machine speed, is limited by the transport of water from the bond zone. Bond strength develops as water is lost from the adhesive. Simulator data show an induction time after initial contact during which the transport producing gradients are established. Increased combining pressure, increased component temperatures, and increased adhesive application temperatures promote wetting and reduce induction times. For the starch based adhesives used in this project, induction times are typically of the order of 5.0 seconds. There is essentially no bond strength development during this time.

Once the induction period ends, the established gradients produce water transport from the adhesive to the components. These transport processes exhibit approximately first order behavior and can be described in terms of a final bond strength (steady-state performance) and a time constant (dynamic performance). For the cold-setting adhesives used in this project the time constant is of the order of 12 seconds. Component temperature is a dominant factor in determining the time constant, but component moisture content, adhesive application rate, component receptivities, and combining pressure play important roles, too. The sketch, in Section II, Part II, page 222, depicts the general form of this bond development function.

Double backer simulator data suggested the following actions to increase double face bonding speeds:

1. Increase adhesive solids levels to reduce the amount of water to be transported.
2. Reduce application rate to the minimum levels consistent with adequate final bond strengths. For the simulator, this level is about 0.5 lb/MSF.
3. Use the maximum possible adhesive application temperature. This, of course, is limited by incipient boiling of the aqueous-based adhesive to 190-195°F.
4. Use the maximum possible combining pressure consistent with board crushing limitations.
5. Preheat the liner.

The simulator also showed that many additives and changes in the adhesive formulation were of no significant value in improving the double face bonding system. Field experience confirmed simulator results.

8. LINER STRETCH IN THE DOUBLE BACKER

Bond slippage, a persistent problem in both the pilot and commercial prototype trials, is exhibited as an apparent relative motion between the flute tip and the liner after initial contact is made. This motion smears the adhesive and reduces the bonded area. The net result is lower bond strength, lower bond effectiveness and reduced bond toughness.

Liner stretch, the cause of bond slippage, was not correctly identified until midway through the commercial prototype trials. As a given flute tip and the corresponding liner segment move through the double backer, the liner is subject to increasing drag forces. The resulting liner stretch obeys a square law because both length and force increase with distance. As the liner stretches, it moves by the flute tip, smearing the adhesive and mechanically interfering with the bond setup process. At the same time, the bond develops strength to carry some of the drag load. Eventually, the bond carries the full load, precluding further stretching. Faster bonding or reduced drag are both effective in reducing the problem.

For more details on liner stretch, including micrographs, see Section IV, Part II, pages 155-161. A micrograph of a hot double facer bond is shown in Fig. 1. Stretch (slippage) is evident in this bond, too. Apparently the magnitude of the slippage in hot corrugating is not sufficient to seriously interfere with bond performance. Nevertheless, it is a factor in reducing bond effectiveness, even in hot corrugating.

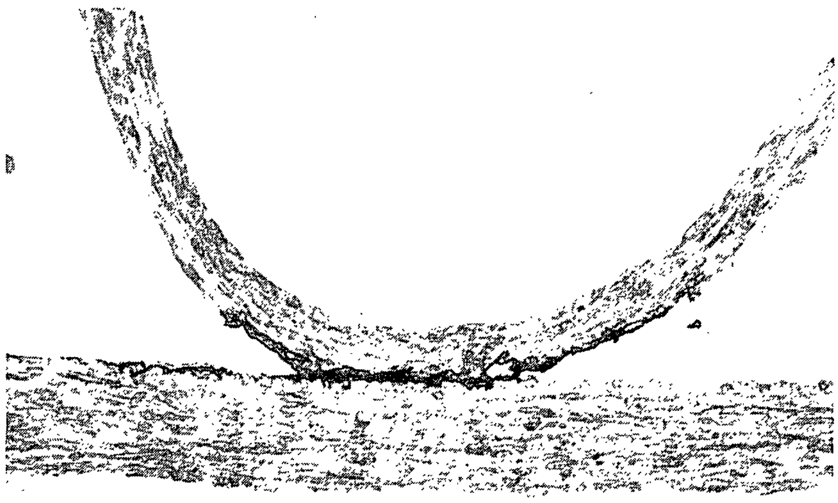

Figure 1. Hot double facer bond.

9. STARCH-BASED SETBACK ADHESIVES

A corrugating bonding system includes the adhesive. the application process, the components and the combining process. In the context of the cold corrugating bonding system, the starch-based, setback adhesive was not totally satisfactory in terms of performance and handling qualities. Nevertheless, this adhesive is capable of producing excellent bonds at very low cost under proper combining conditions. These bonds develop quite rapidly, faster than most commercial cold-setting adhesives tested. Adhesive prepared with pearl starch produces bonds that are water soluble for easy recycling. By substituting a high amylose starch, the bonds may be made water resistant.

The setback adhesive should find application in other areas such as laminating. Added details on this adhesive formulation are contained in Part II of Section II of this report.

10. ADHESIVE APPLICATION AND ROLL STACK DESIGN

The importance of adhesive application control and the difficulty of achieving it are amply documented in various parts of this report. The results show that application rate is extremely important in both SF and DF bonding, that more adhesive may be detrimental in DF bonding, and that adhesive distribution is important in DF bonding, but probably not important in SF bonding.

The banded gravure roll concept, originally used with the permission of the Langston Co., remains a valid concept. The implementation used in this project was inadequate to evaluate the concept. Manufacture of the special roll remains as the important challenge in achieving success with this system. Not enough is known about the release properties of corrugating adhesives, even now, to design the proper patterns.

Increasing adhesive solids levels makes accurate metering more difficult because of the small total amount of material to be handled. The relatively small degree of shear thinning in setback adhesives tends to increase film thickness to gap ratios for two roll applicator systems. This can be partially compensated by low M/A ratios (metering roll surface speed/applicator roll surface speed), but narrow gaps are still required. Gravure rolls and wiper blades meter accurately and consistently, but cannot be varied (hence, the appeal of the banded roll concept).

Fingerless single facers have inherent features that tend to make the application problem more difficult. Because of the vacuum slots or holes in the lower corrugating roll and the absence of finger slots in the top roll, the lower corrugating roll (center roll in the stack) is quite flexible and the top roll is stiff. In fingered machines, the relative flexibilities are reversed. With the stiff roll in the middle of the stack (fingered machine), the two nip loads tend to remain independent and the middle roll straight. With the flexible roll in the middle (fingerless machine), the two nip loads interact through the middle roll, and the middle roll assumes a banana shape. This tends to alter the average gap between the applicator and corrugating roll and to make it also banana shaped in the CD. This can be partially compensated by using a flexible glue roll so it can "conform" to the corrugating roll. However, this "squirming" system is ill-suited to precise and accurate adhesive application. Furthermore, the center portion of the middle roll tends to assume an oval cross section because of the opposing nip loads applied in the roll stack. At the ends, plugs stiffen the roll and keep the cross section circular. This "squashing" of the center part of the roll also gives a nonuniform gap across the machine.

Similar problems are encountered in trying to design for specified, uniform nip loads for forming and bonding.

Despite awareness that roll stack behavior (a consequence of design and operator adjustment) is responsible for nonoptimum adhesive application, fluting and bonding, the industry has not yet developed adequate tools for predicting stack behavior. This is believed to be a primary cause of the many roll stack problems and changeovers experienced in recent years.

In developing tools to analyze the roll stack deficiencies, several useful tools and data were developed. These include relationships between Mullen losses (liner cutting) and pressure roll nip loading (pli) levels, relationships between flute impression depths and pli levels, and so on. A laser dimension gage was used to measure impression depths, but operators should be able to estimate them with sufficient accuracy for setting pressure rolls.

11. WETTED WIDTH SYSTEM

At the end of the project, a special two roll applicator system was installed on the single facer. This system was fitted with a special wetted width control system - see Section IV, Part II, pages 144-146 - which worked extremely well, even with the more challenging setback adhesive. This system maintained a sharply defined, easily adjusted, wetted edge and kept the remainder of the roll completely clean. This system should work very well in conventional adhesive systems.

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SECTION II - THE COLD CORRUGATING PROCESS ELEMENTS

PART I - COLD FORMING

A corrugating process, either hot or cold, has only two process elements: forming or fluting of the corrugating medium and bonding of the medium to the liners. There are two bonding processes: medium to single face liner and medium to double face liner - the first characterized by short duration, high pressure combining; the second by long duration, low pressure combining. Successful cold corrugating requires that the forming and both bonding operations be achieved without heating of the paperboard components. As will be shown, this allows the production of combined board with little or no process heat, less machinery, and less electrical drive energy.

This section of the report will describe the fundamental laboratory studies carried out to develop the basic elements of the cold process. Later sections will translate the results of these studies to pilot plant work and, finally, to the specification and development of a prototype cold corrugating system.

A. COLD FORMING REQUIREMENTS

Cold corrugating requires cold forming (fluting) of the medium. This cold forming process must be workable at full commercial speeds (650 fpm at the time the project was started) and produce a product with end-use performance comparable to that currently available from hot corrugating. All of this must be achieved with regular production corrugating mediums, although there is mounting evidence that modest changes in the medium would greatly improve performance for both hot and cold corrugating.

More specifically, cold forming must

a. work successfully at speeds up to 650 fpm without producing excessive damage or flute fracture,
b. produce typical flute calipers and shapes,
c. produce flutes with height differentials (high-lows) within acceptable commercial limits,
d. produce flutes with structural properties comparable to those produced by the hot forming process, and
e. provide for good release between the formed medium and the corrugating rolls to avoid picking or debris on the corrugating rolls.

Finally, it is necessary that the cold forming process operate within reasonable ranges of machine design and operating parameters, and that it be sufficiently tolerant of operating adjustments to permit realistic commercial production.

All of the above factors have been taken into account in the development of the cold forming process and the machinery to support it. The following parts of this section describe the work completed in understanding the fundamentals of forming and in the approaches to cold forming. Some recent work on improving medium properties is also included.

B. FUNDAMENTALS OF FORMING

1. Flute Fracture - Runnability

Whether formed hot or cold, a corrugating medium always sustains some structural damage. Visible damage is referred to as flute fracture and is indicative of a useless product. The conditions at the onset of fracturing are often used as indicators of runnability. These conditions may relate to the paper, the pretreatment of the paper, or to the machine/process. Less severe damage, i.e., invisible damage, is of importance too and will be treated later in the discussion of structural properties.

Fracture may occur as a bending failure at the outer surface of the flute tip or, more commonly, it may occur as a tensile failure on the flute flank. The latter will be considered first.

In the forming process, the corrugating medium is in contact with several flute tips as shown in Fig. 1. Because of the take-up factor, the medium is drawn into the labyrinth at a speed greater than the surface speed of the corrugating roll. Relative motion between the medium and the flute tips gives rise to a tension force given by:

$$T = T_0 e^{\mu\theta}$$

where T_0 = tension in the free web

μ = coefficient of friction between web and roll

θ = total angle of wrap to point in question

Clearly, the tension value is largest when θ is largest, which corresponds to the center of the labyrinth. This is where tension induced fractures are believed to occur.

Web stiffness or resistance to bending will also contribute to tension buildup and may be a factor in the fracture of heavyweight or very dry mediums. For lightweight or moist mediums, however, friction induced tension is believed to dominate the fracture picture.

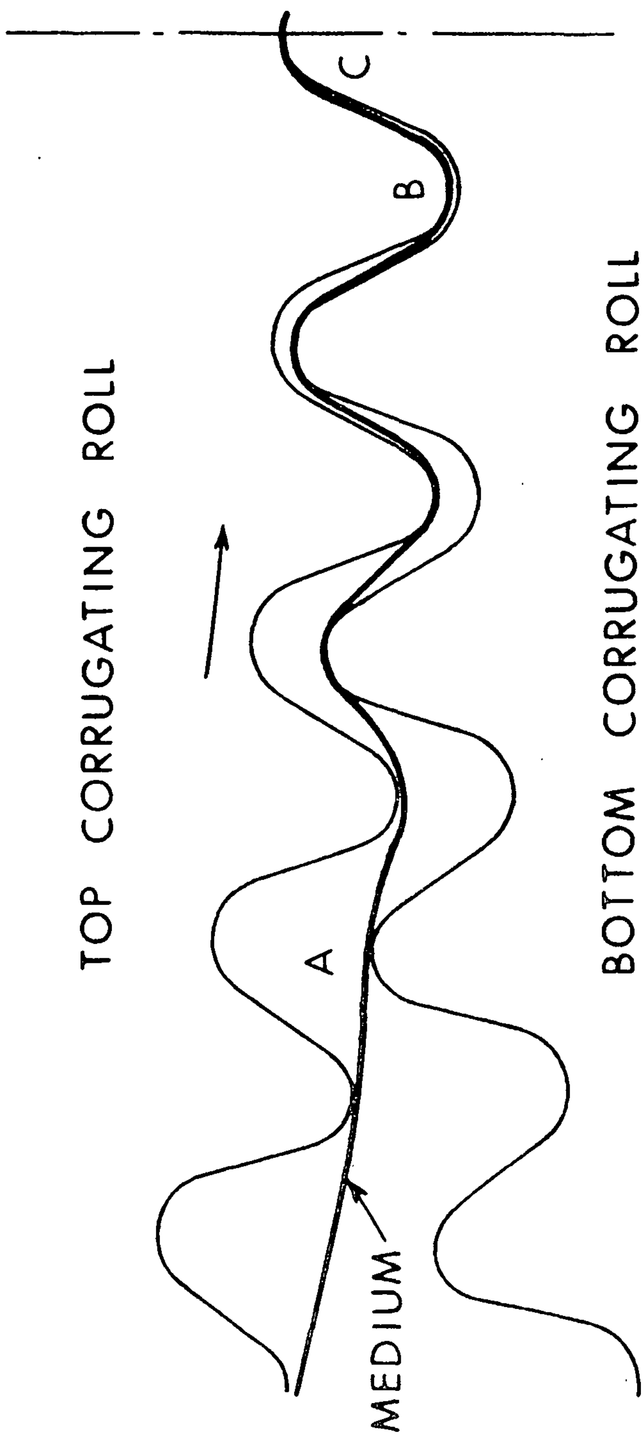

Figure 1. Corrugating roll labyrinth.

To minimize tension build-up and hence the propensity for fracture, one may reduce initial tension T_0, friction coefficient μ, contact angle θ, or all three. Contact angle is a function of flute geometry (flute type) and roll diameter (number of flutes in contact with the web). Smaller rolls are favored for reducing fracture, but other factors impose a practical lower limit. Contact angle may be reduced somewhat by minimizing web wrap around the top corrugating roll. The small contact angle between each flute and the medium makes this a small contribution, however.

Initial tension T_0 may be minimized by using an active tension controlling feeder to deliver the web to the forming nip. Sufficient tension must remain, however, to ensure adequate web tracking. With proper design, such tension control systems can eliminate tension variations due to out-of-round roll stock or other sources and hence the possibility of web fracture due to momentary over-tensions.

To minimize the friction coefficient, the most important contributor to the tension buildup, it is possible to change the corrugating roll surface properties, to change the paper surface properties during manufacture or by pretreatment, or all three. In hot corrugating, friction is reduced by heating the rolls and the medium, by treating one or both with oil mists or sprays, by pretreating the medium with polyethylene, and so on. Presteaming the medium raises moisture content, which generally increases friction but, as will be shown later, generally raises the failure strain limit as well, so that for proper moisture conditioning, the overall tendency to fracture is reduced.

Cold corrugating precludes heating of the rolls and heating and steaming of the medium. Special roll surface properties or pretreatment of either the rolls or the medium to reduce friction are practical, however. Medium pretreatment agents have been developed which are successful in reducing friction and hence the tendency to fracture. Roll surface treatment has received only limited study but has good potential which should be explored further. Recent data suggest that major gains can be made through improved medium properties for both hot and cold forming.

Bending-induced fractures occur because of excessive tensile strain in the outer fibers at the flute tip. In the absence of a shear strain, the outer surface of the medium would have to extend by about 7% to accommodate the flute shape; medium failure occurs at only 3% elongation. Yet failure rarely occurs because shear deformation takes place to reduce the net strain to a value below the failure level. In addition to the bending and shear strains, the medium is also under substantial tension-induced strain and transverse strain from the forming forces (top roll loading). The tension tends to promote failure, whereas the transverse component may actually reduce the tendency to failure. Bending failures are more likely in heavy, dry mediums and the tendency to bending failure increases with increasing tension level. Hence, the mechanisms for reducing tensile fractures reduce the tendency for bending fracture as well. An important additional factor is the medium moisture content at the time of forming.

In summary, the following steps can be taken to minimize the possibility of either type of web fracture during cold forming.

1. Use corrugating rolls with the lowest possible friction coefficient.
2. Avoid wrap of the top corrugating roll by the medium.

3. Prefeed the medium directly into the forming nip under the lowest workable tension.
4. Use the smallest practical roll diameter.
5. Pretreat the medium with a friction reducing agent - low melting point wax is a good example.
6. Run mediums with higher than normal moisture content - 7-8% is a good target.
7. Alter the medium properties to optimize performance.

2. Surface Friction

a. Medium Pretreatment Agents

As was noted in the last section, friction between the medium and the corrugating rolls is an important factor in tension buildup and, hence, to medium damage or fracture. Friction coefficients for corrugating mediums vary widely, and those with a high coefficient are more prone to fracture when formed cold. To alleviate this problem and allow cold forming of virtually all mediums, it is necessary to pretreat at least the high friction mediums with a friction reducing agent. Early in the life of this project considerable effort was devoted to the identification of suitable pretreatment agents. This work is summarized in this section. The following section will briefly consider altering the roll surface properties to achieve the same effect.

In an earlier study for FKBG, various solid "lubricants" were compared for normal corrugating, i.e., at elevated temperature (1). The low to medium density polyethylenes were found to be the most effective agents for normal corrugating. However, preliminary trials indicated that the effectiveness of most agents is different for cold corrugating conditions than for "hot" conditions, as might be expected. Therefore, in the first phase of this study, numerous agents were screened to determine their friction characteristics under room temperature conditions. The best agents, considering both friction and cost, were then selected for trial on the corrugator. Based on the initial corrugator trials, blends of the best agent with other materials were evaluated in order to improve operation on the corrugator. Thus, the development of blends giving the best corrugator results proceeded in a stepwise manner.

i. Friction Screening Tests. Room temperature (73°F) friction tests on candidate pretreatment materials were carried out by abrading a small quantity of each material (prepared as a small cast block) onto a standard medium surface. A specially prepared steel foil was used as the other friction surface. A new foil was used to evaluate each agent to avoid the problem of "cleaning" the metal surface between agent tests. The foils were prepared for test by precleaning the test surface with a piece of sterile cotton saturated with clear acetone. The foils were then held at 350°F for three hours to drive off all traces of residual oils and cooled to room temperature before test.

The lubricant was abraded onto the surface of the medium by applying weights to a cast block of the agent and drawing the medium test strip under the block. To vary the amount of agent applied, three abrading pressures were used; 0.03, 0.17, and 0.83 psi.

After abrading the agent onto the medium, the kinetic coefficient of friction between the foil and the treated surface was measured. Four tests were made to "condition" the foil surface with the agent. Then four tests were made on each of two "standard" medium samples and the results averaged. All tests were carried out under TAPPI Standard conditioning procedures.

ii. Friction Characteristics of Various "Lubricants." A past study for FKBG compared various solid lubricants for normal ("hot") corrugating (1). In general, the low to medium density polyethylenes were found to be the most effective agents under "hot" corrugating conditions. However, exploratory trials suggested that the effectiveness of various lubricating agents is different for cold corrugating conditions than for hot conditions, as would be expected.

Therefore, as an initial step, friction tests were carried out under room temperature (50% RH, 73°F) conditions to evaluate the friction characteristics of various candidate agents. The results are summarized in Table I. It may be noted that many of the agents were evaluated using three abrading pressures in an effort to vary the amount of agent applied to the medium because the friction results appeared to vary somewhat, depending on the amount applied.

The paraffin waxes (see Agents B-2 to B-5 in Table II) exhibited lower friction coefficient at the same abrading pressures than the various polyethylene agents in Table I in these tests at room temperature. In contrast, under "hot" conditions the polyethylene materials gave lower coefficients than the paraffin waxes (1). Under cold conditions these paraffin waxes also exhibited slightly lower friction coefficients than stearin (Agent B-27), which was one of the agents mentioned by Morris and Norman (2).

None of the agents in Tables I and II exhibited a lower coefficient than the paraffin waxes such as Mobilwax 130 (Agent B-3) at the low or intermediate levels of abrading pressure. Therefore, taking cost into consideration, it was decided to use Mobilwax 130 as the base agent in subsequent parts of this study.

TABLE I

KINETIC FRICTION COEFFICIENTS FOR PLASTIC MATERIALS AT STANDARD CONDITIONS

Run No.	Agent	Supplier	Kinetic Friction Coefficients: Ab.Pressure,[a] 0.03 psi	Ab.Pressure,[a] 0.17 psi	Ab.Pressure,[a] 0.83 psi
A-1	Control - untreated medium vs. steel foil		0.57	0.57	0.57
A-2	Polyethylene N-11, density 0.925 g/cc	Eastman Chemical Products Co.	--	0.19	--
A-3	" N-10, " 0.927 g/cc	" " " "	--	0.19	--
A-4	" N-11, " 0.937 g/cc	" " " "	--	0.17	--
A-5	" N-34, " 0.91 g/cc	" " " "	0.17	0.18	0.22
A-6	" M-5W,	" " " "	0.57	0.56	0.52
A-7	" C305G, density 0.86 g/cc	" " " "	--	0.20	--
A-8	" A-C-6, " 0.92 g/cc	Allied Chemical Co.	--	0.22	--
A-9	" A-C-8, " 0.93 g/cc	" " "	--	0.18	--
A-10	Polywax 2000	Petrolite Corporation	--	0.21	--
A-11	Polycarbonate		--	0.46	--
A-12	Polyvinyl chloride		--	0.53	--

[a]Refers to pressure used in abrading agent onto medium surface in order to vary amount applied.

TABLE II

KINETIC FRICTION COEFFICIENTS FOR VARIOUS AGENTS AT STANDARD CONDITIONS

Run No.	Agent	Supplier	Kinetic Friction Coefficients: Ab.Pressure,[a] 0.003 psi	Ab.Pressure,[a] 0.17 psi	Ab.Pressure,[a] 0.83 psi
B-1	Control - untreated medium vs. steel foil		0.57	0.57	0.57
B-2	Mobilwax Db (paraffin wax)	Mobil Oil Corp.	0.12	0.13	0.17
B-3	Mobilwax 130 (paraffin wax, AMP 133-135)	" " "	0.14	0.14	0.18
B-4	Mobilwax 140 (paraffin wax, AMP 143-145)	" " "	0.13	0.14	0.19
B-5	Mobilwax 150 (paraffin wax, AMP 150)	" " "	0.13	0.15	0.23
B-6	Mobilwax 2305 (microcrystalline wax)	" " "	0.15	0.14	0.16
B-7	Amber Be Square 175 (microcrystalline)	Petrolite Corp.	0.23	0.18	0.18
B-8	Amber Be Square 195 (microcrystalline	" " "	0.23	0.18	0.18
B-9	Albaplex (ox. blend microcrystalline wax & PE)	" " "	--	0.23	--
B-10	Oxazoline Wax OWR-150E	Commercial Solvents Corp.	--	0.18	--
B-11	Oxazoline Wax TS970	" " "	--	0.17	--
B-12	Oxazoline Wax TS970AA	" " "	--	0.21	--
B-13	Advawax 140 (fatty acid ester)	Cincinnati Milacron Chem. Co.	--	0.23	--
B-14	Advawax 165 (refined paraffin lub.)	" " " "	--	0.17	--
B-15	Advawax 225 (fatty amide wax)	" " " "	--	0.17	--
B-16	Advawax 240 (fatty amide wax)	" " " "	--	0.16	--
B-17	Advawax 290 (bisstearamide-type wax)	" " " "	--	0.21	--
B-18	Beeswax	--	0.17	0.14	0.12
B-19	Carbowax 4000	Union Carbide	--	0.17	--
B-20	Acrawax C	Glyco Chemicals Inc.	--	0.21	--
B-21	Paraflint RG (mixture straight chain paraffins)	Moore & Munger	--	0.18	--
B-21	Armoslip O (oleamide)	Armak	0.17	0.21	0.43
B-22	Armoslip CPM (oleamide)	"	0.16	--	--
B-23	Armoslip 18 (stearamide)	"	0.38	0.37	0.22
B-24	Armoslip HT (hydrogenated tallow amide)	"	0.15	--	--
B-25	Glycon S-70 (70% stearic acid)	Glyco Chemicals Inc.	0.17	0.14	0.12
B-26	Glycon S-80 (80% stearic acid)	" " "	0.25	0.17	0.14
B-27	Glycon HTG (stearin, hydrogenated tallow glyceride)	" " "	--	0.23	--
B-28	Graphite (Microfyne type)	The Jos. Dixon Crucible Co.	0.20	0.17	0.16

[a]Refers to pressure used in abrading agent onto medium surface in order to vary amount applied.
[b]No longer made; Mobilwax 130 is similar product.

Some of the initial exploratory tests were carried out with Mobilwax D, but for later tests this obsolete material was replaced with Mobilwax 130, an equivalent product.

To determine if additional reductions in friction could be obtained, blends of Mobilwax 130 with various agents were evaluated as shown in Table III.

Blends with various types of graphite in quantities up to 5% were prepared and tested - see Agents C-4 to C-11. In general, modestly lower friction coefficients were obtained with the Microfyne or equivalent graphite product No. 1110 at the 1 to 5% addition level, particularly at the high abrading pressure. The reduction in friction coefficient obtained with the wax-graphite blends may not be statistically significant; however, the initial corrugating trials, to be discussed later in the text, appeared to confirm that the addition of 1% of graphite No. 1110 increased the maximum runnability speed in terms of fractured flutes.

The addition of stearin in amounts up to 50% did not appear to give any improvement in the friction characteristics relative to the base agent alone, i.e., Mobilwax 130 or Mobilwax D. However, later corrugator trials indicated that the addition of stearin in amounts of 5% or more tended to reduce or prevent "picking" of the medium in the corrugating roll nip for mediums exhibiting this tendency.

Other organic blending agents investigated included stearic acid, oleamide, stearamide, Polywax, carnauba wax, and Cerax 1320, a carnauba wax substitute supplied by the F. B. Ross Company. At the given addition level, blending agents did not give any major change in friction relative to the

TABLE III

KINETIC FRICTION COEFFICIENTS FOR BLENDS OF VARIOUS AGENTS WITH PARAFFIN WAX AT STANDARD CONDITIONS

Run No.	Blend Composition	Kinetic Friction Coefficients: Ab.Pressure,[a] 0.03 psi	Ab.Pressure,[a] 0.17 psi	Ab.Pressure,[a] 0.83 psi
C-1	Control - untreated medium (Samples A and B) vs. steel foil	0.57	0.57	0.57
C-2	" - Mobilwax 130	0.14	0.14	0.18
C-3	- Mobilwax D	0.12	0.13	0.17
C-4	99% Mobilwax 130 + 1% gaphite (Microfyne) (Jos. Dixon Crucible Co.)	0.12	0.11	0.11
C-5	98% " " + 2% " " " " " "	0.12	0.11	0.12
C-6	95% " " + 5% " " " " " "	0.13	0.12	0.11
C-7	99% " " + 1% flake graphite (Jos. Dixon Crucible Co.)	0.13	0.11	0.11
C-8	99% " " + 1% Dixon graphite KS-2	0.14	0.14	0.17
C-9	99% " " + 1% " " KS-5	0.13	0.14	0.17
C-10	99% " " + 1% " " KS-10	0.13	0.14	0.17
C-11	99% " " + 1% " " 1110	0.11	0.11	0.13
C-12	99.5% " " + 0.5% stearin (Glycon HTG)	0.13	0.15	0.19
C-13	99% " " + 1% " " "	0.13	0.14	0.17
C-14	95% " D + 5% " " "	0.13	--	--
C-15	50% " D + 50% " " "	--	0.16	0.21
C-16	99.5% " 130 + 0.5% stearic acid (Glycon S-70)	0.13	0.13	0.17
C-17	99% " " + 1% " " " "	0.15	0.13	0.15
C-18	95% " D + 5% " " " "	0.14	--	--
C-19	99.5% " 130 + 0.5% " " (Glycon S-80)	0.18	0.14	0.15
C-20	99% " " + 1% " " " "	0.13	0.14	0.18
C-21	90% " " + 10% PE N-11 (Eastman Chemical Co.)	0.13	0.14	0.20
C-22	80% " " + 20% " " " " "	0.15	0.16	0.20
C-23	95% " " + 5% talc (Mistron Vapor, Cyprus Industrial Minerals Co.)	0.12	0.13	0.16
C-24	90% " " + 10% " " " " " " "	0.13	0.13	0.14
C-25	80% " " + 20% " " " " " " "	0.12	0.12	0.14
C-26	60% " " + 40% " " " " " " "	0.12	0.13	0.15
C-27	90% " " + 10% " " HGO-55, Cyprus Industrial Minerals Co.)	0.16	0.13	0.16
C-28	80% " " + 20% " " " " " " "	0.16	0.13	0.15
C-29	95% " " + 5% pearl cornstarch (Cargill)	0.12	0.12	0.12
C-30	90% " " + 10% " " "	0.15	0.13	0.13
C-31	80% " " + 20% " " "	0.12	0.12	0.13
C-32	60% " " + 40% " " "	0.14	0.12	0.11
C-33	99% " " + 1% Armoslip O (oleamide, Armak)	0.14	0.12	0.14
C-34	99% " " + 1% " CPM (oleamide, Armak)	0.12	0.12	0.15
C-35	99% " " + 1% " 18 (stearamide, Armak)	0.12	0.14	0.16
C-36	99% " " + 1% No. 200 Silicone (Dow Corning)	0.18	0.18	0.18
C-37	99% " " + 1% Dow F 157 wax (Dow Corning)	0.14	0.15	0.18
C-38	99% " " + 5% Polywax 2000 (Petrolite Corporation)	0.14	0.13	0.17
C-39	95% " " + 5% "	0.13	0.12	0.16
C-40	95% " " + 5% No. 3 NC carnauba wax (F. B. Ross Company)	0.12	0.13	0.18
C-41	94% " " + 5% Cerax No. 1320 (F. B. Ross Company)	0.12	0.15	0.22

[a]Refers to pressure used in abrading agent onto medium surface in order to vary amount applied.

Mobilwax 130 alone. However, subsequent corrugator trails indicated that Cerax 1320 at a 5% addition level tended to reduce "picking" with certain mediums as in the case of stearin.

The addition of talc in amounts ranging from 5 to 40% was also investigated because of its low cost. As may be noted in Table III, the friction results obtained with the wax-talc blends tended to be slightly lower than obtained with wax alone, although the differences in friction were probably not significant.

Based on the above, it was decided to initiate actual cold forming trials using the following agents.

1. Mobilwax 130 (MW 130)
2. 99% Mobilwax 130 + 1% graphite (No. 1110)
3. 80% Mobilwax 130 + 20% talc (Mistron Vapor)

b. Corrugating Roll Surface Treatments

The fundamental mechanics of the corrugating process and the experimental results cited in the previous section clearly show the importance of minimizing friction between the medium and the corrugating rolls. Medium pretreatment agents are applied to the medium surface to reduce the friction forces, but agents apparently transfer to the corrugating rolls as a part of the friction reducing process. As a result, the effect is retained for several hundred feet of additional operation after the treatment of the medium is discontinued. To be effective, such pretreatment must be applied at essentially all times.

An alternative to continuous pretreatment of the corrugating medium is a one-time treatment of the corrugating roll surface to give a permanent reduction in the friction coefficient. For a given roll, the actual friction coefficient will depend on the specific roll finishing processes and materials, but none of the options currently available in roll manufacture is expected to suffice for cold forming untreated mediums. Recently, however, a new permanent surface treatment process became available which should reduce friction and improve release while retaining roll life. Such a surface should improve runnability and reduce fracture or flute damage, two important ingredients in cold forming.

The new surface is developed by the TFE-LOK® process (34) which permanently locks a large number of small bits of Teflon in a chrome surface layer. This gives the surface the friction characteristics of Teflon and the wear characteristics of a chrome surface. Two mediums, with friction coefficients against steel of 0.57 and 0.27, respectively, were tested against a TFE-LOK surface. In both cases, the friction coefficient was 0.16. In previous work, it was shown that mediums with a friction coefficient of less than 0.4 could be corrugated without pretreatment. Hence, the TFE-LOK process shows promise of obviating the pretreatment step.

The TFE-LOK process requires a uniform chrome layer with a thickness of at least 1.5 mils. To provide a set of rolls for test, we commissioned a roll rebuilding company to alter the profiles on a set of A-flute rolls to accommodate the necessary chrome and provide rolls with the required plating. They experienced great difficulty in delivering a satisfactory product, and the final profiles were not suitable for running. The TFE-LOK process was carried out without difficulty and the surface exhibited the expected low friction levels. Because of the poor flute profiles, however, no useful test results were developed.

Because of the pressures of other issues, the existence of a workable cold forming process, and the unavailability of a set of "expendable" rolls, no further evaluation of the TEF-LOK process was carried out. It remains as a potentially very useful approach to eliminating pretreatment and minimizing some of the release and contamination problems discussed later. It may also have application in hot corrugating.

c. Dual Corrugating Roll Drives

Flute fracture is the most dramatic type of forming deficiency and one which cannot be tolerated in any form. It is believed to be primarily a tension failure resulting from excessive tension buildup in the forming operation. Medium treatment agents reduce the drag and shear forces on the medium and, hence, the tension loads. Other mechanisms for reducing tension are also available. These include reducing the wrap on the top corrugating roll via an idler roll at the nip, feeding the web directly to nip under the low, but controlled, tension, and treating the corrugating rolls to reduce friction. Results from early work on some of these approaches were presented above. Tension may also be induced by the transverse loads on the medium caused by transmitting the torque required to drive the top roll through the flute flanks. If so, supplying the drive torque directly to the top roll rather than through the medium should improve performance. This concept was tested by building a dual drive for the single facer.

In this system, the corrugator drive motor is connected to a drive shaft through a multiple V-belt drive. This shaft drives the lower corrugating roll through a single-stage gear reduction. Connected to this same shaft is a gear box with proper ratios and drive sense to drive the top roll at the same speed as the bottom roll. The output shaft of the gear box is connected to the top roll through two gear couplings and a shaft. This permits the necessary up and down motion of the top roll. Inside the gear box is a mechanical phase adjuster which can be set so the bottom roll provides all the torque for the top roll or vice versa, or anywhere in between. The adjustment to the gear box is external and can be changed while the corrugator is running.

To evaluate the dual drive system, single-face board samples were prepared and examined for changes in draw factor, caliper, flat crush, high-lows, etc., as a function of the forming process configuration and adjustment. These samples were prepared using MTA's to avoid any unnecessary problems with flute fracture.

While minor variations existed, it was very difficult to discern any significant trends. From these test results, it was concluded that under the range of forming process parameters tested, the dual drive was not effective in improving draw factor, caliper or flat crush. Because of these results and the pressure of other important issues, no further attention was given to the dual drive concept.

3. Cold Forming Development - Single Facer Trials

a. Pretreatment Agent Application Methods

For cold forming trials on the laboratory single facer, "lubricating" agents were abraded onto both sides of the medium at a station just before the medium preconditioner, as illustrated in Fig. 2. The lubricant was cast into bars about 12 inches long by 1.25 inches wide by 1.75 inches deep as shown in Fig. 3. The bars were cast with slots on each side near the top (see Fig. 3) so that they could be easily inserted and removed from the holders on the machine.

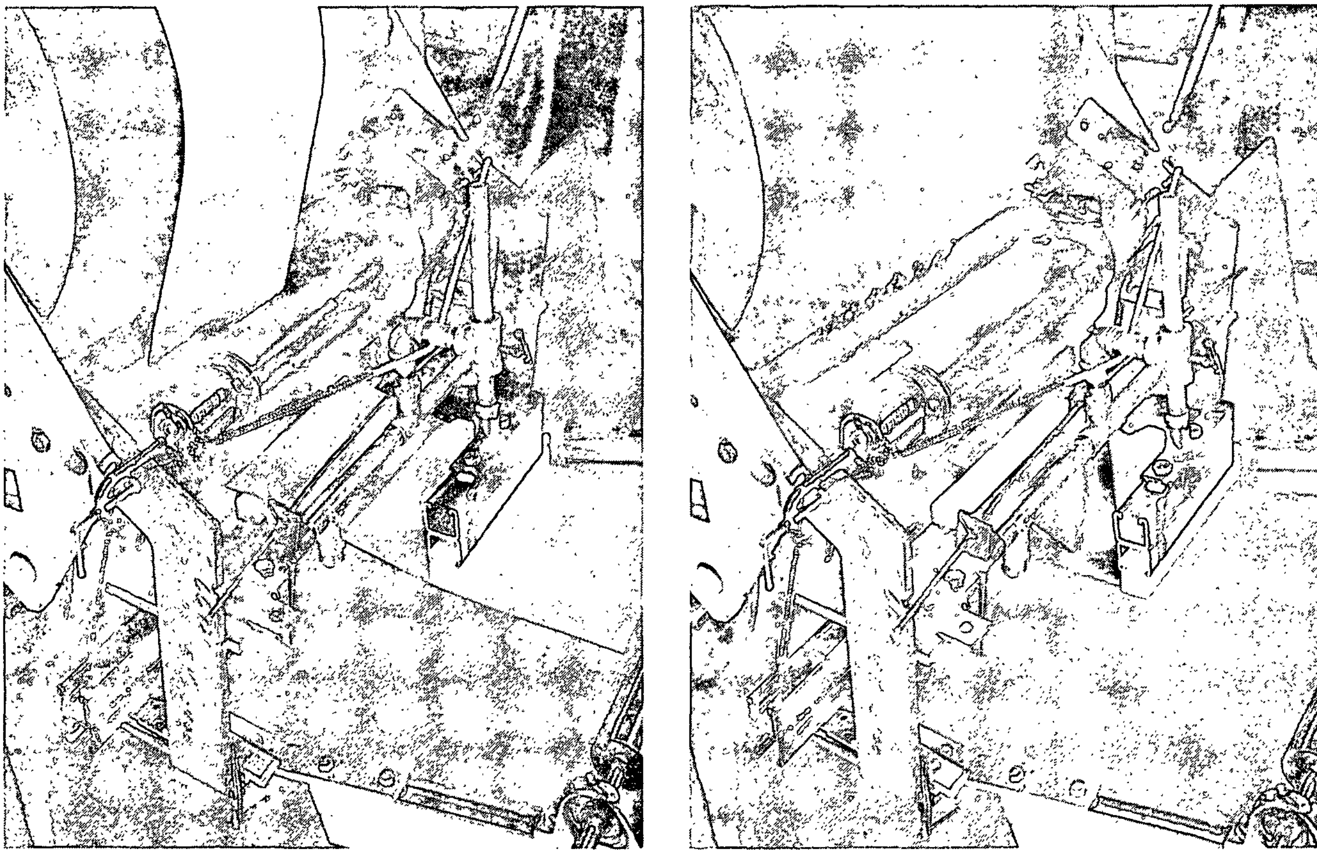

Figure 2. "Lubricant" application system.

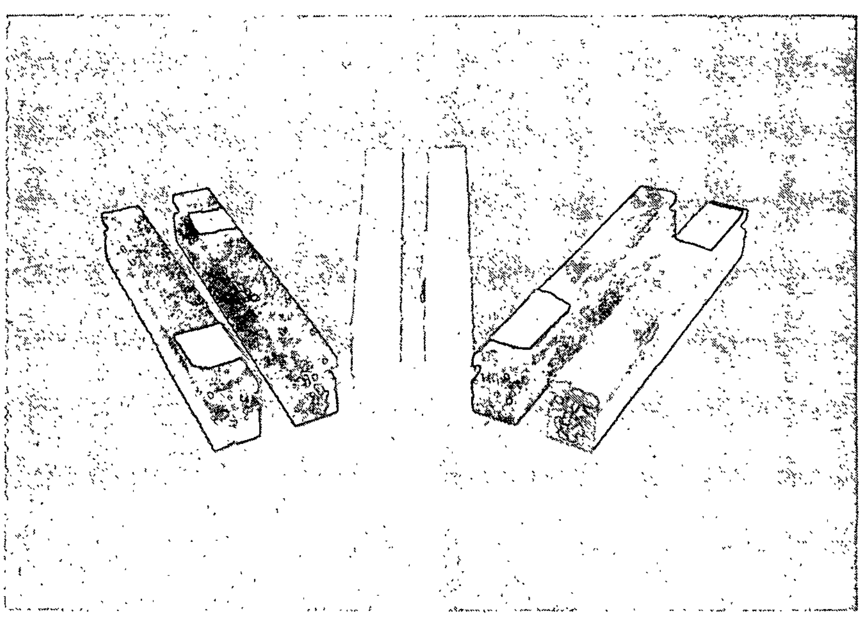

Figure 3. Appearance of cast lubricant bars.

As shown in Fig. 2, the upper and lower agent bars were offset in the direction of web travel by about 8 inches. In an initial trial, the bars were set opposing each other so the medium web passed through a nip formed by the two bars. With this arrangement, it was possible to vary the amount of agent applied by weighting the arms holding the lubricant. However, with the rectangular bar configuration used, high spots in the medium tended to catch in the nip between the lubricant bars and tear the web. Therefore, the offset bar arrangement was employed in all the trials except the first. With this arrangement, application rate can be controlled by varying the degree of loading of the individual bars.

b. Evaluation of Treatment Agents in Cold Forming Trials

As outlined above, friction tests were used to identify pretreatment agents with low friction coefficients. When used in corrugating, these agents were expected to reduce the tension buildup and hence the degree of damage or fracture of the corrugating medium. To further evaluate this concept and these materials, limited forming trials were conducted by operating the single facer without adhesive, thus leaving the liner and formed medium unbonded. Treatment agents were applied by abrasion, as described above. Five typical corrugating mediums were used for these trials, and the corrugator was operated without any steam, i.e., at room temperature.

The single facer used for all laboratory trials is a special experimental unit built by Langston. Because the working faces of the rolls are narrow (12 inch paper is used) the rolls are not crowned. All tests were conducted without pressure roll stops and with A-flute rolls. This is an older machine with fixed fingers, either fluff or nonfluff.

For some early single facing trials, a polyvinyl acetate adhesive was used to bond the formed medium to the liner.

Forming results for the above three agents are shown in Table IV. In addition, results obtained using a blend of polyethylene (Eastman N-11) and polyethylene glycol dioleate, a blend used in past work under "hot" corrugating conditions, are included for comparison. Because the components were not bonded it was difficult to handle the corrugated medium at speeds above 400 fpm. Therefore, except for Runs 5A and 5B, the corrugating speed was increased up to 400 fpm at 0.5 lb/inch web tension. If fracturing was not observed under these conditions, the web tension was increased in 0.5 lb/inch increments until fracture occurred.

As noted in the table, all of these test mediums fractured at "idle" speed when fluted without pretreatment. For pretreatment with these agents the best runnability, in terms of web tension at 400 fpm, was obtained with the blend of Mobilwax 130 and 1% graphite. This agent also showed the lowest consumption level, and the cost of the agent was estimated to be less than about $0.18 per 1000 sq ft of single-faced board, i.e., $1.80 per million square feet of board.

Following the preliminary cold forming trials described above, additional trials were conducted using small amounts of shower steam (Lodding-type showers) to determine if improved molding would result. The initial trials in this series suggested that the shower steam would be beneficial. However, some of the test mediums tended to "pick" out in the corrugating rolls under these conditions, indicating a need for modification of the lubricant used (Mobilwax

TABLE IV

COMPARISON OF LUBRICATING AGENTS FOR "COLD" CORRUGATING

Run	Corrugating Medium (26 lb)	No Lubricating Agent	Application Force, lb/inch of width	Type of Lubricating Agent: Mobil-wax 130	Blend 1	Blend 2	Blend 3
		Maximum runnability condition, speed fpm at indicated tension lb/inch					
1A	A (northern semichem.)	Idle @ 0.5	0.5	400 @ 1.5[a]	400 @ 2.0[a]	400 @ 1.0[a]	100 @ 0.5[a]
B			0.9	400 @ 2.5[a]	--	--	--
2A	B (southern semichem.)	Idle @ 0.5	0.5	400 @ 2.0[a]	400 @ 3.0[a]	400 @ 0.5[a]	100 @ 0.5[a]
B				--	--	--	400 @ 0.5
3	C (southern semichem.)	Idle @ 0.5	0.5	400 @ 2.5	400 @ 3.5+	400 @ 2.5	400 @ 0.5
4	D (southern semichem.)	Idle @ 0.5	0.5	400 @ 3.5+	400 @ 3.5+	400 @ 3.5+	400 @ 0.5
5A	E (southern semichem.)	Idle @ 0.5	0.5	600 @ 1.5[a]	--	--	--
B			0.9	600 @ 1.5[a]	--	--	--
Average maximum tension @ 400 fpm and 0.5 lb/inch application force				2.4+	3.0+	1.9	0.5
Estimated lubricant consumption, lb/M ft^2 of single-faced board				0.029	0.014	0.035	0.016

[a]Slight decapping noted at all speed and tension conditions.

Note: Because of difficulty in manually handling the corrugated unlined medium, the maximum speed employed was 400 fpm except for Runs 5A and B. Thus, in Runs 1-4 the speed was increased in increments up to 400 fpm and 0.5 lb/inch increments up to fracture or the maximum obtainable tension (3.5 lb/inch).

130 + 1% graphite). To this end the following modified blends were evaluated on the corrugator to determine their effectiveness in reducing picking:

1. 95% Mobilwax 130, 5% stearin
2. 94% Mobilwax 130, 5% stearin, 1% graphite
3. 50% Mobilwax 130, 50% stearin - a blend mentioned in the Morris and Norman patent (4)
4. 94% Mobilwax 130, 5% Cerax 1320 (a carnauba wax substitute), 1% graphite.

For these and subsequent trials, the medium was bonded to the liner to permit evaluation of both runnability and single-face board properties. These samples were evaluated for the following properties:

1. flat crush, 5 tests per corrugator speed level
2. single-faced ring compression, 5 tests per corrugator speed level
3. flute height, 50 determinations per corrugator speed level
4. average difference in flute height, 45 determinations per corrugator speed level.

The single-faced ring compression specimens were 1.25 inches wide by 8.70 inches long. The specimens were formed into cylinders 1.25 inches high, and the vertical joint was taped with 60-lb paper tape. After forming, the specimens were edge dipped in wax on both ends. The formed and dipped cylinders were tested in an H&D tester.

The single-faced board materials were preconditioned for at least 24 hours at less than 35% RH, 73°F and conditioned for at least 48 hours at 50% RH and 73°F prior to test.

The runnability results obtained are shown in Table V. No "picking" was encountered except in one instance with blend No. 1 above. All of the blends permitted high-speed corrugating without fracture in the case of 26-lb mediums. However, the results for the 26-lb northern semichemical medium (Runs B2-B5) indicated that the blends with 1% graphite gave higher runnabilities in terms of either maximum speed or web tension in accord with previous results. For the same runs the 50/50 blend of wax and stearin gave a lower maximum runnability than the 94/5/1 blends of wax/stearin/graphite or wax/Cerax 1320/graphite.

For the 33-lb medium the maximum speed under cold conditions was 400 fpm as compared to 500 fpm under hot conditions. This suggested that further modifications of the lubricant blend would be desirable to improve operation with heavyweight mediums.

The lubricant consumptions in these tests ranged from about 3 to 14 lb per million sq ft of single-faced board. The consumption levels varied considerably, depending on the medium characteristics and lubricant blend. Based on the cost of the ingredients and consumption levels, the costs varied from about $0.53 to $2.60 per million sq ft of single-faced corrugated board.

The properties of the single-faced boards obtained with the above agents under cold conditions are shown in Table VI. For each medium, samples were evaluated under both normal (hot) and cold conditions at two corrugating speeds - generally 300 and 500 fpm. Composite averages are also shown for the four lubricant blends, based on the results for the 26-lb northern and southern semichemical mediums.

TABLE V

CORRUGATING TRIALS USING LUBRICATING AGENT BLENDS OF WAXES, STEARIN AND GRAPHITE

Run	Type of Medium	Corr Roll Temp.	Lubricant Type[a]	Lubricant Amount,[b] lb/M ft2	Lubricant Cost,[c] $/M ft2	Runnability Speed, fpm	Runnability Web Tension, lb/inch	Power[d] Volts	Power[d] Amperes
A-1	26-lb Southern semichem.	350°F	--	--	--	500	2.0	110	55
A-2	" " "	Cold	W5S	9.55	1.24	500	3.5+	110	52
A-3	" " "	Cold	W5SG	7.89	1.05	500	3.5+	115	54
A-4	" " "	Cold	W50S	4.54	0.95	500	3.5+	110	51
A-5	" " "	Cold	W5CG	9.10	1.57	500	3.5+	110	50
B-1	26-lb Northern semichem.	350°F	--	--	--	500	1.5	110	54
B-2	" " "	Cold	W5S	7.06	0.92	300[e]	0.5[e]	110	54
B-3	" " "	Cold	W5SG	--	--	500	2.5	110	52
B-4	" " "	Cold	W50S	12.4	2.60	500	1.5	115	51
B-5	" " "	Cold	W5CG	25.3	2.45	500	2.5	--	--
C-1	26-lb Reclaimed	350°F	--	--	--	500	3.5+	110	54
C-2	" "	Cold	W5SG	--	--	500	3.5+	110	49
C-3	" "	Cold	W5CG	3.09	0.53	500	3.5+	110	49
D-1	33-lb Southern semichem	350°F	--	--	--	500	0.5	110	60
D-2	" " "	Cold	W5SG	12.5	1.66	400	0.5	115	58
D-3	" " "	Cold	W5CG	12.6	2.18	300	0.5	115	58

[a]Lubricant Code
W5S - 95% Mobilwax 130 + 5% stearin
W5SG - 94% Mobilwax 130 + 5% stearin + 1% graphite
W50S - 50% Mobilwax 130 + 50% stearin
W5CG - 94% Mobilwax 130 + 5% Cerax 1320 + 1% graphite.
[b]Amount in lb per million square feet of single-faced board.
[c]Cost in dollars per million square feet of single-faced board.
[d]Power readings at 500 fpm.
[e]Picking occurred preventing running at higher speeds.

TABLE VI

EFFECT OF TYPE OF LUBRICANT ON SINGLE-FACED BOARD PROPERTIES
UNDER COLD CORRUGATING CONDITIONS

Run	Type of Medium	Corr. Roll Temp., °F	Lubricant[a]	Speed, fpm	Single-faced Board Characteristics: Flat Crush, psi	Pin Adhesion, lb	S.F. Ring Comp., lb/inch	Flute Height, pt	Av. Height Diff., pt
A-1	26-lb Southern semichem.	350	--	300	27.7	89.0	34.6	193.2	2.08
				500	29.3	85.2	34.9	193.7	2.51
				Av.	28.5	87.1	34.8	193.4	2.30
A-2	" " "	<100	W5S	300	24.4	87.2	38.6	199.5	4.81
				500	25.8	86.6	38.0	201.2	4.04
				Av.	25.1	86.9	38.3	199.8	4.42
A-3	" " "	<100	W5SG	300	23.8	89.2	37.0	201,4	4.45
				500	24.4	90.8	36.8	202.5	3.68
				Av.	24.1	90.0	36.9	202.0	4.06
A-4	" " "	<100	W50S	300	23.6	81.8	37.8	201.1	3.32
				500	24.0	86.2	35.6	201.5	5.00
				Av.	23.8	84.0	36.7	201.3	4.16
A-5	" " "	<100	W5CG	300	24.0	84.4	37.2	201.6	2.67
				500	22.6	85.2	36.7	202.2	4.24
				Av.	23.3	84.8	37.0	201.9	3.46
B-1	26-lb Northern semichem.	350	--	300	32.5	71.8	34.9	194.7	1.02
				500	32.8	71.2	34.0	194.2	1.99
				Av.	32.6	71.5	34.4	194.4	1.50
B-2	" " "	<100	W5S	300	28.1	81.0	37.7	198.4	2.97
				500	27.7	83.2	36.3	198.9	3.36
				Av.	27.9	82.1	37.0	198.6	3.16
B-3	" " "	<100	W5SG	300	27.8	81.2	38.6	200.2	2.80
				500	27.2	84.4	36.7	201.6	3.01
				Av.	27.5	82.8	37.6	200.9	2.90
B-4	" " "	<100	W50S	300	27.3	87.4	37.2	200.5	3.53
				500	27.8	82.4	36.4	201.4	3.28
				Av.	27.6	84.9	36.8	201.0	3.40
B-5	" " "	<100	W5CG	300	27.8	83.2	37.4	201.2	1.98
				500	27.2	86.8	35.7	202.2	3.11
				Av.	27.5	85.0	36.6	201.7	2.54

See end of table for footnote.

TABLE VI - CONTINUED

EFFECT OF TYPE OF LUBRICANT ON SINGLE-FACED BOARD PROPERTIES
UNDER COLD CORRUGATING CONDITIONS

Run	Type of Medium	Corr. Roll Temp., °F	Lubricant[a]	Speed, fpm	Single-faced Board Characteristics: Flat Crush, psi	Pin Adhesion, lb	S.F. Ring Comp., lb/inch	Flute Height, pt	Av. Height Diff., pt
C-1	26-lb Reclaimed	350	--	300	26.6	85.6	28.7	193.3	1.73
				500	25.3	78.0	27.8	194.5	1.67
				Av.	26.0	81.8	28.2	193.9	1.70
C-2	" "	<100	W5SG	300	21.6	86.0	29.6	197.4	2.75
				500	21.0	89.4	30.5	200.1	2.69
				Av.	21.3	87.7	30.0	198.8	2.72
C-3	" "	<100	W5CG	300	22.2	89.4	30.5	197.6	3.09
				500	20.7	90.0	30.3	198.9	2.43
				Av.	21.4	89.7	30.4	198.2	2.76
D-1	33-lb Southern semichem.	350	--	300	46.4	83.0	37.6	196.1	1.66
				500	47.3	71.8	36.6	196.4	2.85
				Av.	46.8	77.4	37.1	196.2	2.26
D-2	" " "	<100	W5SG	300	37.2	79.4	39.9	202.7	2.63
				400	36.4	80.0	38.1	202.7	2.24
				Av.	36.8	79.7	39.0	202.7	2.44
D-3	" " "	<100	W5CG	100	35.4	81.0	40.3	199.5	1.84
				300	34.4	91.6	34.8	202.1	2.38
				Av.	34.9	86.3	37.6	200.8	2.11
Composite - 26-lb Northern and southern semichem. at 300 and 500 fpm		350	--	--	30.6	79.3	34.6	193.9	1.90
		<100	W5S	--	26.5	84.5	37.6	199.2	3.79
		<100	W5SG	--	25.8	86.4	37.2	201.4	3.48
		<100	W50S	--	25.7	84.4	36.8	201.2	3.78
		<100	W5CG	--	25.4	84.9	36.8	201.8	3.00

[a]Lubricant Code
W5S - 95% Mobilwax 130, 5% stearin
W5SG - 94% Mobilwax 130, 5% stearin, 1% graphite
W50S - 50% Mobilwax 130, 50% stearin
W5CG - 94% Mobilwax 130, 5% Cerax 1320, 1% graphite.

The cold corrugated board exhibited lower flat crush by 3-5 psi in the case of the 26-lb northern and southern semichemical mediums. (Note: In later trials smaller differences in flat crush were observed after the single-faced boards were made into double-faced board.) In contrast, the cold corrugated boards exhibited higher single-faced ring compression and flute height values. The increase in single-faced ring compression would be expected to improve top-load box compressive strength.

The average (flute) height differences shown in Table VI are a measure of the tendency to form high-lows. In general the cold corrugated board exhibited somewhat higher flute height differences than the normal (hot) corrugated board. Thus, it appeared that modification of the composition of the lubricant and changes in the corrugator operating conditions would be desirable as well.

Along these lines, additional exploratory trials suggested that the addition of about 1% of Dow silicone oil No. 200 to the wax/stearin/graphite blend would help reduce high-lows and improve the runnability of the 33-lb medium. Other work indicated that higher top corrugating roll pressure tended to reduce high-lows. Accordingly, a series of corrugating trials was carried out to compare the effectiveness of the following lubricant blends:

1. 94% Mobilwax 130, 5% stearin, 1% graphite - Code W5SG
2. 93% Mobilwax 130, 5% stearin, 1% graphite, 1% silicone oil No. 200 - Code W5SG1S
3. 93% Mobilwax 130, 5% Cerax 1320, 1% graphite, 1% silicone oil No. 200 - Code W5CG1S.

The cold corrugating trials with these agents were carried out using a 25% higher top corrugating roll pressure. The results obtained are summarized in Table VII. As in previous trials, the amount of lubricant used was quite small - ranging from about 4 to 22 lb/million sq ft. The estimated costs of the agents, based on the measured application quantities, ranged from about $0.53 to $3.62 per million sq ft, depending on the medium and the blend.

The results in Table VII show that the cold corrugated boards made with the blends containing silicone oil generally exhibited high-low levels which were about equal and, in some cases, better than the high-low levels achieved in the normal (hot) corrugating trials. Thus, it appears that commercially acceptable high-low levels can be obtained under cold conditions.

The cold corrugated single-faced boards also exhibited higher flute heights and single-face ring compression strength than the boards made under hot conditions. The cold corrugated boards exhibited lower flat crush strengths than the hot corrugated boards, as was noted in previous trials.

In this connection, a number of single-faced board samples were manually made into double-faced board and evaluated for caliper, flat crush and CD edgewise compression. The results are shown in Table VIII. After double-backing, the flat crush levels for the cold corrugated boards made with 26-lb medium were roughly comparable to the levels obtained on the hot corrugated boards. However, in the case of the 33-lb medium the flat crush of the cold corrugated board was 12.9% lower than for the "hot" corrugated board. The cold corrugated boards, after double-backing, also tended to exhibit slightly higher caliper and CD edgewise compression strengths.

Reservations have been expressed with regard to the use of silicone oil because it may migrate during storage and affect bonding. However, at the low

TABLE VII

COMPARISON OF THE EFFECTIVENESS OF VARIOUS LUBRICANTS FOR COLD CORRUGATING CONDITIONS

Run No.	Type of Medium	Corr. Roll Temp., °F	Lubricant Type[a]	Lubricant Amount,[b] lb/M ft²	Lubricant Cost,[b] $/M ft²	Speed, fpm	Single-faced Board Characteristics: Flat Crush, psi	S.F. Ring Comp., lb/inch	Flute Height, pt	Av. Height Diff., pt
A-1	26-lb Southern semichem.	350	--	--	--	500	29.3	34.9	193.7	2.51
SP-9	" " "	Cold	W5SG	4.0	0.53	500	27.6	38.1	199.3	4.00
SP-7	" " "	Cold	W5SG1S	6.0	0.94	500	26.4	37.7	200.0	2.64
SP-6	" " "	Cold	W5CG1S	4.6	0.91	500	26.1	36.0	198.9	2.87
B-1	26-lb Southern semichem.	350	--	--	--	500	32.8	34.0	194.2	1.99
B-11	" " "	Cold	W5SG	--	--	500	28.1	36.8	197.1	2.71
SP-11	" " "	Cold	W5SG1S	14.4	2.26	500	27.2	35.2	201.6	1.58
SP-10	" " "	Cold	W5CG1S	15.3	3.01	500	27.5	35.4	201.5	1.98
C-1	26-lb Reclaimed	350	--	--	--	500	25.3	27.8	194.5	1.67
C-2	" "	Cold	W5SG	--	--	500	21.0	30.5	200.1	2.69
SP-13	" "	Cold	W5SG1S	10.6	1.66	500	22.8	31.0	198.8	2.12
SP-12	" "	Cold	W5CG1S	4.6	0.91	500	22.4	31.0	198.6	1.80
D-1	33-lb Southern semichem.	350	--	--	--	500	47.3	36.6	196.4	2.85
SP-14	" " "	Cold	W5SG	22.1	2.94	500	36.8	37.8	202.6	1.26
SP-17	" " "	Cold	W5SG1S	--	--	500	37.6	35.5	203.7	2.24
SP-16	" " "	Cold	W5CG1S	18.4	3.62	400	37.0	38.3	202.6	1.96

[a]Lubricant Code:
W5S - 95% Mobilwax 130, 5% stearin, 1% graphite
W5SG1S - 93% Mobilwax 130, 5% stearin, 1% graphite, 1% silicone oil.
W5CG1S - 93% Mobilwax 130, 5% Cerax 1320, 1% graphite, 1% silicone oil.
[b]Amount and cost in units per million square feet of single-faced board.

Note: Speeds were limited to 500 fpm in this phase by loss of adhesion with the polyvinyl acetate adhesive used.

TABLE VIII

COMPARISON OF COMBINED BOARD CHARACTERISTICS

Run No.	Type of Medium	Corr. Roll Temp., °F	Lubricant	Double-faced Board Characteristics					
				Caliper, pt	Diff., %	Flat Crush, psi	Diff., %	CD Edgewise Compression, lb/inch	Diff., %
A-1	26-lb Southern semichem.	350	None	192.9	--	28.9	--	56.1	--
SP-7	" " "	< 100	W5SG1S	194.4	+0.8	29.3	+1.4	58.3	+3.9
B-1	26-lb Northern semichem.	350	None	191.2	--	30.4	--	54.4	--
SP-11	" " "	< 100	W5SG1S	193.6	+1.3	28.3	-6.9	55.0	-1.1
C-1	26-lb Reclaimed	350	None	193.0	--	26.4	--	46.9	--
SP-13	" "	< 100	W5SG1S	195.6	+1.3	26.2	-0.8	51.1	+9.0
D-1	33-lb Southern semichem.	350	None	197.0	--	42.8	--	57.0	--
SP-17	" " "	< 100	W5SG1S	200.2	+1.6	37.3	-12.9	59.8	+4.9

levels of application in cold corrugating this effect may not be noticeable. For example, the following pin adhesion strengths were obtained on single-faced board from Run SP-7 after various storage times at 100°F:

	Pin Adhesion, lb	Change, %[a]
1. Initial test average	83.8	
2. After 4 weeks storage	84.0	+0.2
3. After 8 weeks storage	83.0	-1.0
4. After 12 weeks storage	87.8	+4.8

[a]Based on initial results as reference.

Thus, little or no change in pin adhesion occurred in the 12-week period with the PVAc adhesive used in these trials.

c. Effect of Operating Conditions

A series of trials was carried out to determine the effect of various corrugator operating conditions on the characteristics of single-faced board made under cold corrugating conditions. The effects of increasing the top corrugating roll pressure on single-faced board characteristics are shown in Table IX.

Referring to the table it may be noted that the 26-lb southern semi-chemical medium was corrugated using top corrugating roll pressures of 327, 409, and 490 pli, corresponding to standard, +25 and +50% pressures, respectively. Trials were made for this medium using two steam shower conditions; no shower steam and low shower steam. The results indicated that the main effect of the higher top roll pressure was to decrease the tendency to form high-lows, i.e.,

TABLE IX

EFFECT OF TOP CORRUGATING ROLL PRESSURE ON COLD CORRUGATED SINGLE-FACED BOARD CHARACTERISTICS

Run No.	Type of Medium	Corr. Roll Temp., °F	Corr. Roll Pressure, pli	Steam Shower Condition	Single-faced Board Characteristics[a] Flat Crush, psi	S.F. Ring Comp., lb/inch	Pin Adhesion, lb	Flute Height, pt	Av. Height Diff., pt
A-1	26-lb Southern semichem.	350	327	Normal	28.5	34.8	87.1	193.4	2.30
A-3	" " "	<100[b]	327	None	24.1	36.9	90.0	202.0	4.06
A-17	" " "	<100[b]	409	None	25.2	38.0	--	199.9	3.35
A-18	" " "	<100[b]	490	None	25.4	38.6	--	200.0	3.01
A-13	26-lb Southern semichem.	<100[b]	327	Low	25.8	38.6	--	198.0	4.36
A-20	" " "	<100[b]	409	Low	25.7	37.9	87.6	198.8	2.13
A-21	" " "	<100[b]	490	Low	25.3	37.1	86.0	198.8	2.60
B-1	26-lb Northern semichem.	350	327	Normal	32.6	34.4	71.5	194.4	1.50
B-9	" " "	<100[b]	327	Low	28.1	37.4	--	199.4	3.00
B-11	" " "	<100[b]	409	Low	28.2	36.8	--	197.8	3.06
C-1	26-lb Reclaimed	350	327	Normal	26.0	28.2	81.8	193.9	1.70
C-7	" "	<100[b]	327	Low	22.8	32.8	--	199.4	2.64
C-9	" "	<100[b]	409	Low	22.1	32.4	--	198.7	2.14
D-1	33-lb Southern semichem.	350	327	Normal	46.8	37.1	77.4	196.2	2.26
D-7	" " "	<100[c]	327	Low	38.0	41.4	--	203.8	2.00
D-9	" " "	<100[c]	409	Low	37.6	42.0	--	203.1	1.64
Composite (26-lb medium)		350	327	Normal	29.0	32.5	80.1	193.9	1.83
"		<100	327	Low	25.6	36.3	--	198.9	3.33
"		<100	409	Low	25.3	35.7	--	198.4	2.44

[a]Average of results at 300 and 500 fpm.
[b]The lubricant composition was 94% Mobilwax 130, 5% stearin, 1% graphite.
[c]The lubricant composition was 89% Mobilwax 130, 5% Cerax 1320, 1% graphite, 5% silicone oil.

reduce the average (flute) height differences. For this medium the lowest flute height differences were obtained with the top roll pressure increased by 25% in conjunction with a low amount of shower steam.

Based on the above results, the other mediums were tested at two pressure levels; standard and +25% (409 pli). The higher top corrugating roll pressure reduced high-lows for the 26-lb reclaimed and 33-lb semichemical medium. The high top roll pressure did not have any appreciable effect on the high-lows obtained with the 26-lb northern semichemical medium.

Taken as a whole, these results indicate that higher top corrugating roll pressure is advantageous under cold conditions. Thus, changes in roll crown may be desirable for cold corrugating.

A series of trials was carried out to determine if the application of shower steam to the web prior to fluting would improve the molding of the flute. Because the web was at room temperature, two steam shower pressures were used to avoid overmoistening of the web. The results are shown in Table X. The "low" level of shower application resulted in slight increases in flat crush and single-face ring compression - apparently due to improved molding. When the shower pressure was increased to the "high" level, little or no further improvement in these properties was obtained with the 26-lb mediums. In the case of the 33-lb medium, operation with the "high" shower steam level resulted in fracturing at 100 fpm which could not be overcome with the lubricant used. In general, the use of the showers did not appear to decrease the occurrence of high-lows.

TABLE X

EFFECT OF STEAM SHOWER CONDITION ON COLD CORRUGATED SINGLE-FACED BOARD CHARACTERISTICS

Run No.	Type of Medium	Corr. Roll Temp., °F	Corr. Roll Pressure, pli	Steam Shower Condition[a]	Single-faced Board Characteristics[b] Flat Crush, psi	S.F. Ring Comp., lb/inch	Pin Adhesion, lb	Flute Height, pt	Av. Height Diff., pt
A-1	26-lb Southern semichem.	350	327	Normal	28.5	34.8	87.1	193.4	2.30
A-3	" " "	<100[c]	327	None	24.1	36.9	90.0	202.0	4.06
A-13	" " "	<100[c]	327	Low	25.8	38.6	--	198.0	4.36
A-15	" " "	<100[c]	327	High	25.5	37.8	--	197.2	4.42
B-1	26-lb Northern semichem.	350	327	Normal	32.6	34.4	71.5	194.4	1.50
B-3	" " "	<100[c]	327	None	27.5	37.6	82.8	200.9	2.90
B-9	" " "	<100[c]	327	Low	28.1	37.4	--	199.4	3.00
B-10	" " "	<100[c]	327	High	29.0	37.4	--	197.4	2.94
C-1	26-lb Reclaimed	350	327	Normal	26.0	28.2	81.8	193.9	1.70
C-2	" "	<100[c]	327	None	21.3	30.0	87.7	198.8	2.72
C-7	" "	<100[c]	327	Low	22.8	32.8	--	199.4	2.64
C-8	" "	<100[c]	327	High	23.8	32.8	--	198.5	2.40
D-1	33-lb Southern semichem.	350	327	Normal	46.8	37.1	77.4	196.2	2.26
D-2	" " "	<100[c]	327	None	36.8	39.0	79.7	202.7	1.84
D-7	" " "	<100[d]	327	Low	38.0	41.4	--	203.8	2.00
D-8	" " "	<100[d]	327	High	--[e]	--[e]	--[e]	--[e]	--[e]
Composite (26-lb medium)		350	327	Normal	29.0	32.5	80.1	193.9	1.83
"		<100	327	None	24.3	34.8	86.8	200.6	3.23
"		<100	327	Low	25.6	36.3	--	198.9	3.33
"		<100	327	High	26.1	36.0	--	197.7	3.25

[a]Lodding showers used in cold corrugating runs. Low steam pressures were employed.
[b]Average of results at 300 and 500 fpm.
[c]The lubricant composition was 94% Mobilwax 130, 5% stearin, 1% graphite.
[d]The lubricant composition was 89% Mobilwax 130, 5% Cerax 1320, 1% graphite, 5% silicone oil.
[e]Fractured at 100 fpm.

During these and other trials it appeared that "picking" difficulties increased if excessive localized moistening occurred, e.g., if condensate dripped on the medium web.

Table XI compares the effect of using relief and no-relief fingers on the characteristics of the single-faced board. At the standard top corrugating roll pressure (327 pli) the board made with no-relief fingers exhibited less tendency to form high-lows (lower flute height difference) than the board made with relief-type fingers. The other board properties remained about the same. For the runs made with higher top corrugating roll pressures the characteristics the single-faced board made with the two finger-types were approximately the same. The results are quite limited but might indicate that no-relief fingers have an advantage in reducing high-lows, under cold conditions using "normal" top roll pressures.

d. Effect of Medium Moisture

A limited series of trials was carried out to investigate the effects of medium moisture content on the characteristics of the single-faced board. To vary the medium moisture content, one roll of 26-lb southern semichemical medium was run through a water spray to elevate its moisture content. The wet web was then subjected to three levels of drying during the rewinding operation. By this means it was planned to obtain rewound rolls having three levels of moisture in the 2 to 11% range. However, the actual levels obtained were 4.0, 9.2, and 18.9%. While the latter value was considerably higher than desired, it was decided to proceed with the corrugating trials. The results obtained at 300 fpm are summarized in Table XII. For corrugating without shower steam, flat crush strength increased slightly with moisture content, but the flute height decreased substantially at the highest moisture content. The high-lows tended to be a

TABLE XI

COMPARISON OF RELIEF AND NO-RELIEF FINGERS UNDER COLD CORRUGATING CONDITIONS

(26-lb Southern semichemical medium)

					Single-faced Board Characteristics[b]				
Run No.	Type of Finger	Corr. Roll Temp., °F	Corr. Roll Pressure, pli	Steam Shower Condition[a]	Flat Crush, psi	S.F. Ring Comp., lb/inch	Pin Adhesion, lb	Flute Height, pt	Av. Height Diff., pt
A-13-1	Relief	<100	327	Low	25.8	38.6	--	198.0	4.36
A-13-2	No-relief	<100	327	Low	26.0	35.4	--	198.4	2.90
A-20-1	Relief	<100	409	Low	25.7	37.9	--	198.8	2.13
A-20-2	No-relief	<100	409	Low	25.0	36.2	--	198.4	2.64
A-21-1	Relief	<100	490	Low	25.3	37.1	--	198.8	2.60
A-21-2	No-relief	<100	490	Low	25.2	36.1	--	199.2	2.58

[a]Lubricant composition: 94% Mobilwax 130, 5% stearin, 1% gaphite.

TABLE XII

EFFECT OF MEDIUM MOISTURE ON COLD CORRUGATED BOARD

(26-lb Southern semichemical medium)

Run[a]	Medium Moisture, %	Steam Shower Condition	Corr. Roll Temp., °F	Single-faced Board Characteristics at 300 fpm: Flat Crush, psi	S.F. Ring Comp., lb/inch	Flute Height, pt	Av. Height Diff., pt	Pin Adhesion, lb
26	4.0	None	<100	25.0	39.0	203.3	2.73	64.8
27	9.2	None	<100	26.0	38.2	198.7	2.02	75.4
28	18.9	None	<100	28.3	38.0	191.1	2.84	88.4
29	4.0	Low	<100	26.1	39.6	202.1	2.42	72.4
30	9.2	Low	<100	25.8	38.6	198.1	2.47	77.0
31	18.9	Low	<100	26.8	37.3	189.8	2.57	91.8

[a]Lubricant composition: 94% Mobilwax 130, 5% stearin, 1% graphite.

minimum at the intermediate moisture level - 9.2%. When shower steam was employed, the board properties were essentially the same at the three medium moisture levels except that (1) the flute height was low at the 18.9% medium moisture level and (2) the pin adhesion strength increased with increasing medium moisture content. The pin adhesion strengths also increased with increasing medium moisture when no shower steam was used. The trend to higher pin adhesion strength may merely reflect differences in adhesive transfer at the various medium moisture levels with the PVAc adhesive used in these trials. In any case, it appears that the cold corrugating process can be employed with mediums exhibiting commercial moisture content tolerances, although more work would be required to determine optimum levels.

C. STRUCTURAL PERFORMANCE AND ANALYSIS

The work described above was essential in the early development of a cold forming process. However, in that work only limited attention was paid to the structural performance of cold-formed flutes. More recent fundamental work on the basic fluting process shows great potential for improving performance and the utilization of fiber. The basic relationships between medium properties and forming performance had not been previously defined, even in the case of hot corrugating; this lack was even more evident in cold corrugating. There is evidence that medium properties can be optimized with respect to cold forming; however, it appears that this can best be accomplished by basic studies of the forming process. Defining the medium properties required for good formability and structural performance was undertaken to guide modification of the manufacturing process at the corrugating medium mill to produce mediums with the optimum balance of properties.

Knowledge gained from fundamental research on cold forming would have had immediate application as the cold process was implemented by various box plants. However, it also has immediate application to the hot process, now operating on 600-700 corrugators. Thus, while this work was initially directed to optimizing the medium for cold forming, the results can be used to improve the industry's use of fiber and energy in the hot process.

Accordingly, the overall objective of this research was to analyze the corrugating forming process to determine the characteristics which optimize the structural performance of the board, while maintaining or improving runnability under cold forming conditions. This includes consideration of hot forming where appropriate to basic understanding. Therefore, initial emphasis was placed on determining how medium and board performance is affected by cold and hot fluting. However, the results show that the losses incurred in both fluting processes are much more important than the differences between hot and cold fluting. Hence, these losses, rather than the differences between hot and cold forming, were the primary target of this research.

In the first phase of this work attention was directed to the effects of forming conditions, forming geometry and flat crush behavior.

1. Background

Most of the published work on the corrugating process is concerned with hot corrugating. However, many of the forming phenomena are similar in hot and cold forming. Therefore, the forming concepts discussed below draw heavily on the hot corrugating literature, but care has been taken to make distinctions between hot and cold forming as seem appropriate.

In general, corrugating performance under either cold or hot fluting conditions is limited by several factors which affect board quality. They include flute fractures or damage, high-lows and adhesion. For example, under hot conditions, fractured flutes lower the quality of the board, as noted by McKee and Gander (3). They reported losses in flat crush ranging up to 13% and losses in edgewise compressive strength of up to 20%. Similar losses can occur under cold conditions. Usually the strength falls off gradually with increasing speed because only sporadic fractures are initially encountered, as noted by Gottsching and Otto (13) in their work on the hot process. However, the presence of fractures is usually marked by an increase in flat crush variability. Thus, the corrugating process imposes high stresses in the medium which can lower board quality if they exceed the "strength" of the medium. Even though fracture does not occur, the stresses during fluting reduce the strength of the fluted medium. These effects of the fluting stresses are the subject of this work.

At the entrance to the labyrinth (see Fig. 1) the main stress on the medium is due to the applied brake tension. The tensile stress at A is approximately constant across the medium thickness. The web tension in the medium is one of the most important factors affecting runnability and board quality. Many investigators have shown that, for the hot process, increasing web tension lowers the speed at which fracture occurs (3, 13-15). Our work has shown that increasing web tension has a similar effect in cold corrugating.

In the labyrinth (regions A to C, Fig. 1) the medium must travel faster than the tips on the corrugating rolls to accommodate the take-up or draw. As a result, there is a frictional drag on the medium. The drag progressively increases the web tension as the medium moves toward the center of the forming nip. McKee and Gander (3,4) showed that the web tension near the center of the labyrinth is much greater than the initial or brake tension. Figure 4 shows that the web tension in the final stages of forming is strongly dependent on the coefficient of friction, μ, between the medium and the corrugating rolls. Recently, Thomas (5) obtained similar results in an analysis of the C-flute labyrinth (Fig. 5).

Figure 5 shows that increasing the coefficient of friction between the medium and corrugating roll surfaces from 0.2 to 0.3 will cause a 100% or more increase in the final tension in the medium. For this reason the application of a small amount of certain "slip" agents to the medium or corrugating roll will reduce the tension in the medium during forming. As noted earlier, markedly higher corrugating speeds can be achieved using such agents. The effectiveness of various agents under "hot" corrugating conditions is discussed in Ref. (6). "Slip" agents also reduce the tendency to form high-lows under hot conditions (6,7).

Under cold corrugating conditions, we have found that many mediums must be treated with friction reducing agents to prevent fracture and minimize high-lows (8-10). The coefficient of friction of corrugating medium is often much higher at room temperature than under hot corrugating conditions. In such cases flute fracture can occur unless suitable agents are used. Later in this report we (4) summarize the results of experimental corrugating trials on 35 commercial mediums. The mediums were evaluated to determine runnability and board quality both with and without application of medium treatment agents. Almost all mediums were successfully cold formed at commercial speeds and web tensions by using treatment agents; those mediums with friction coefficients below about 0.4 (about

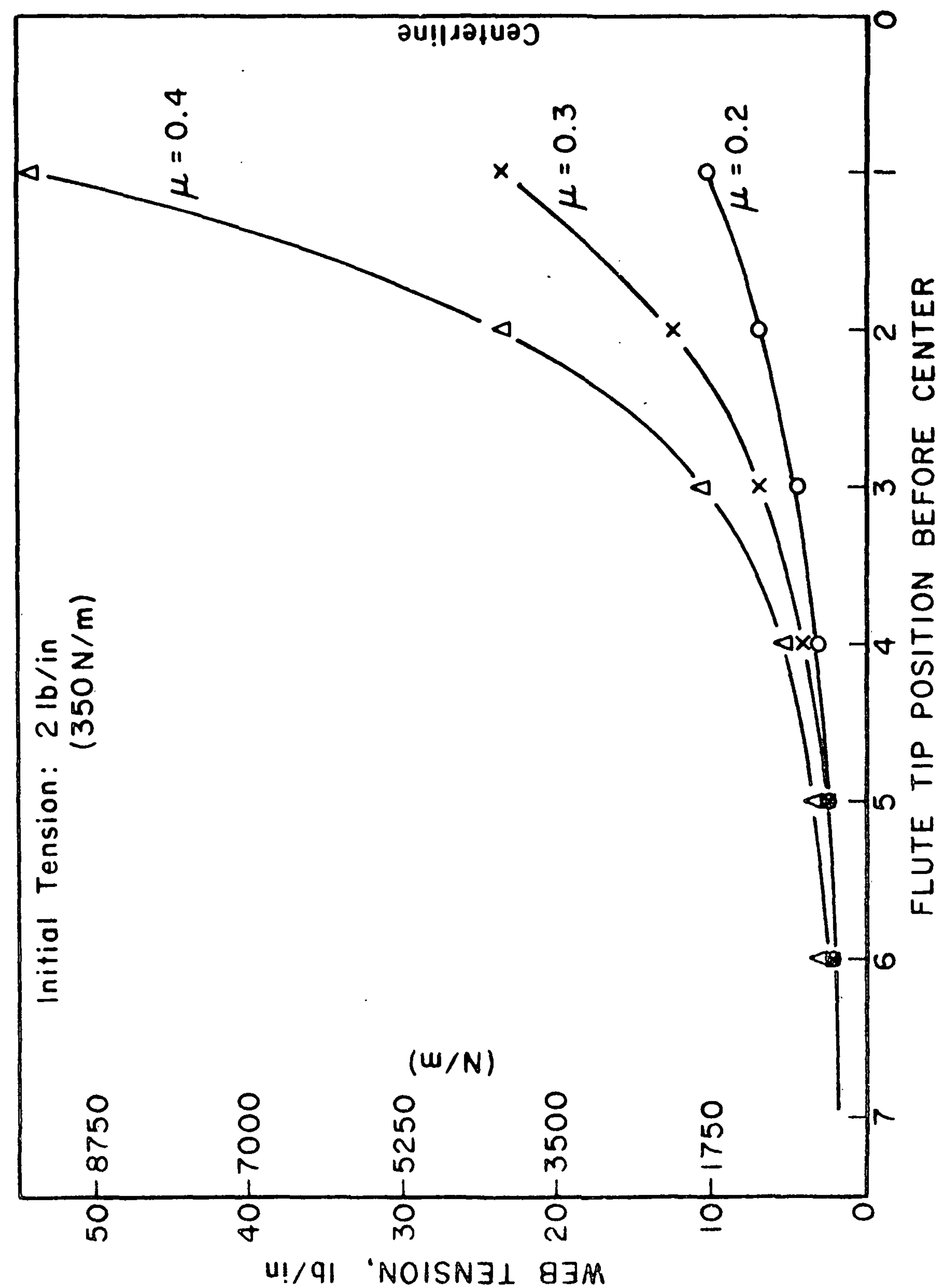

Figure 4. Effect of friction on web tensions in the corrugating labyrinth (μ = coefficient of friction) [From Ref. (9)].

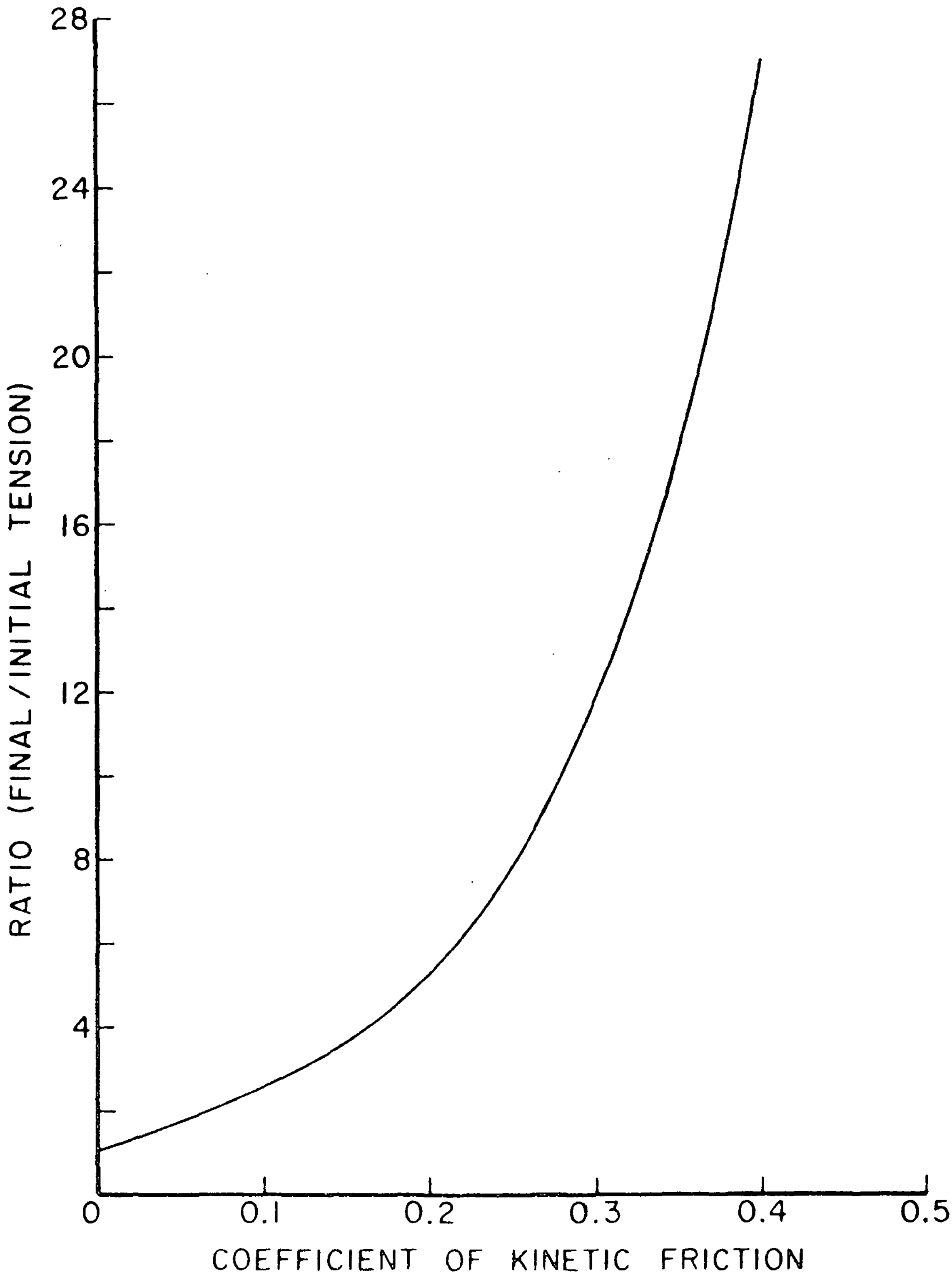

Figure 5. Maximum web tension in labyrinth for a C-flute contour [from Ref. (10)].

half of all mediums tested) were successfully cold formed at commercial speeds without treatment agents. Our results also indicate that the effectiveness of various slip agents and hence the choice of a proper agent varies with the corrugating temperature condition (8).

The medium at the flute tips is bent to the tooth radius, giving rise to tensile stresses on the outside and compressive stresses on the inside (Fig. 6B). When added to the transport tensions (Fig. 6A), the stress on the outside convex surface is increased as illustrated in Fig. 6C. A critical state of stress may be reached when the transport and bending stresses are added and hence cause tip fracture. The bending stresses and strains will depend on the medium caliper and radius of curvatures of the corrugating roll flute tip. The higher the caliper or the smaller the radius, the greater the strain and hence likelihood of fracture.

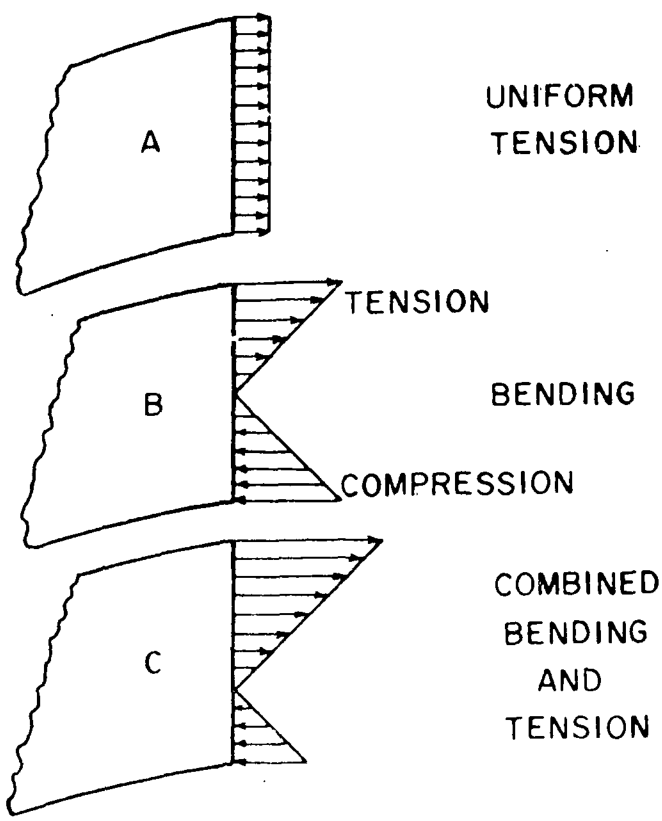

Figure 6. Combined bending and web (uniform) tension [Ref. (9)].

Estimates of bending strain on the flute tip are greater than the allowable machine direction stretch of medium (3,5); however, shear stresses induced in the medium during forming reduce the net bending strain to permit forming without fracture. The transverse shear modulus of medium is quite low compared to the in-plane moduli, thus permitting high shear strain levels at low shear stress levels. This is due to the "layered" nature of most paperboards. The shear strains generated in the medium will reduce the intensity of the bending strains and assist in the forming. As an extreme example, some mediums delaminate during hot corrugating due to excessively high shear strains; the same may occur under cold conditions, although it has not been observed in the mediums tested to this time.

At the center of the labyrinth, point C in Fig. 1, high transverse compressive forces are applied to the medium. The medium thickness in the tip and root regions is reduced, which helps "set" the flute contour (3). Much greater caliper reductions are obtained for hot corrugating than for cold corrugating.

The web tension and transverse compressive stresses oscillate in magnitude during the formation of each flute due to the up-and-down motion of the top corrugating roll (11,12) and possible draw variations in the labyrinth. The stress oscillations occur in hot forming and are believed to also occur in cold forming. Figure 7 illustrates the oscillatory nature of the web tension, top corrugating roll pressure and "up-and-down" corrugating roll acceleration under hot conditions (12). The fundamental frequency of the oscillating forces is the flute forming frequency, but large higher harmonics are usually present. The variations in web tension are particularly important because they will be magnified in the labyrinth. Substantial cyclic peaks in web tension could occur as a result.

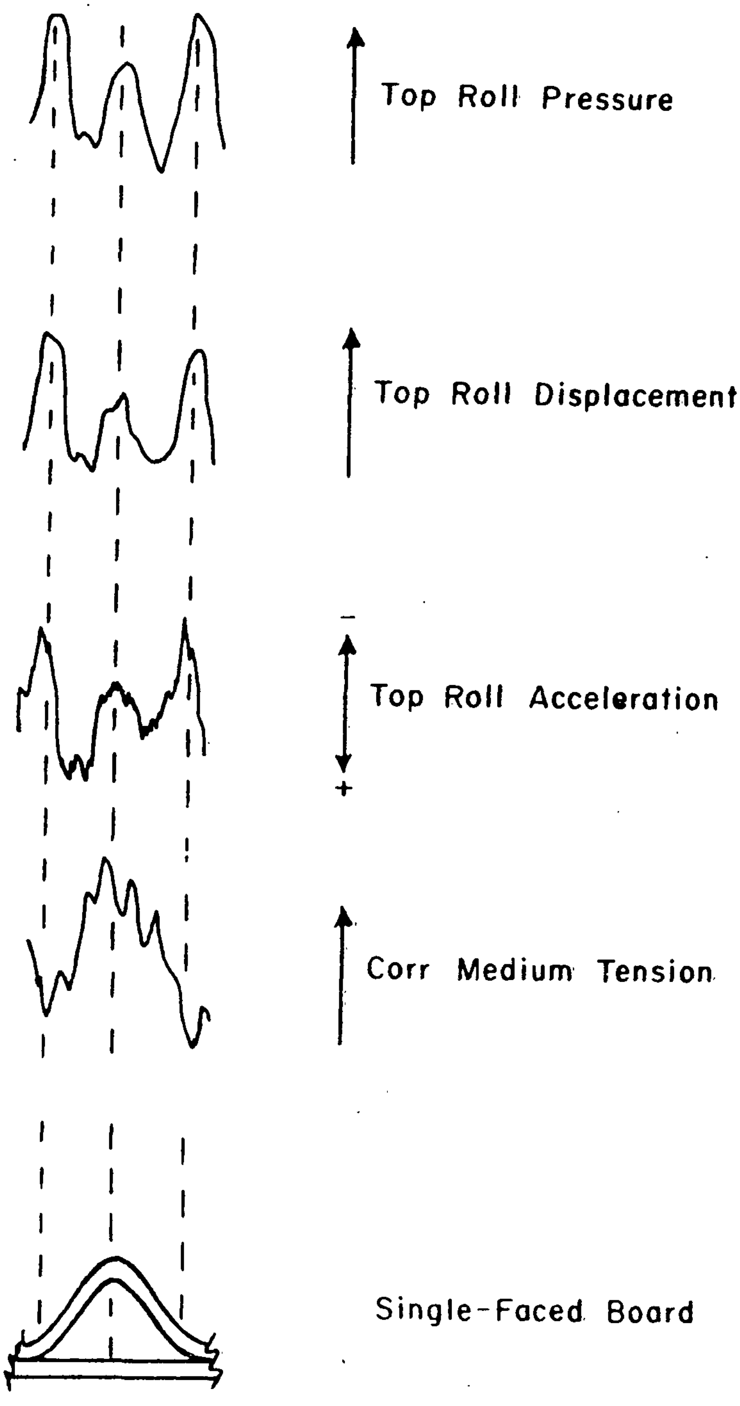

Figure 7. Oscillatory nature of corrugating forces and corrugating roll displacement [Ref. (18)].

a. Temperature and Moisture Effects in Corrugating

Various authors have emphasized the importance of corrugating temperature on both flute fracturing and high lows [see Ref. (13-18)]. It is believed that the lignin and hemicellulose components in the medium become more plastic and "flow" at high corrugating temperatures, particularly if somewhat moist. The "flow" of these components is believed to assist the medium in forming to the flute contour and in retaining its flute shape so as to minimize high-low flute formation in the hot process. This hypothesis explains many of the phenomena observed in normal corrugating with hot (350°F) corrugating rolls. However, we have shown that corrugating can be carried out under room temperature conditions where thermal softening effects do not occur. It also may be remarked that the new fingerless corrugators have reduced high-low problems (19-21) under hot conditions and presumably will do so under cold conditions.

b. Cold *vs*. Hot Corrugating Flat Crush

Most commercial mediums can be corrugated satisfactorily under cold conditions. In general, the properties of the cold formed board are approximately the same as those obtained under hot corrugating conditions. However, cold formed board made with some mediums exhibits lower ultimate flat crush strength than hot formed board. While the ultimate flat crush strengths may differ, the initial portions of the load-deflection curves are comparable for cold and hot formed board (Fig. 8). The first peak strengths are approximately equal for all commercial mediums evaluated to date. Thus, cold and hot formed board should show about the same response when subjected to low degrees of crushing by pull rolls and belts.

When combined board is crushed between rigid steel rolls, all boards (hot or cold formed) will suffer the same amount of caliper reduction. Medium

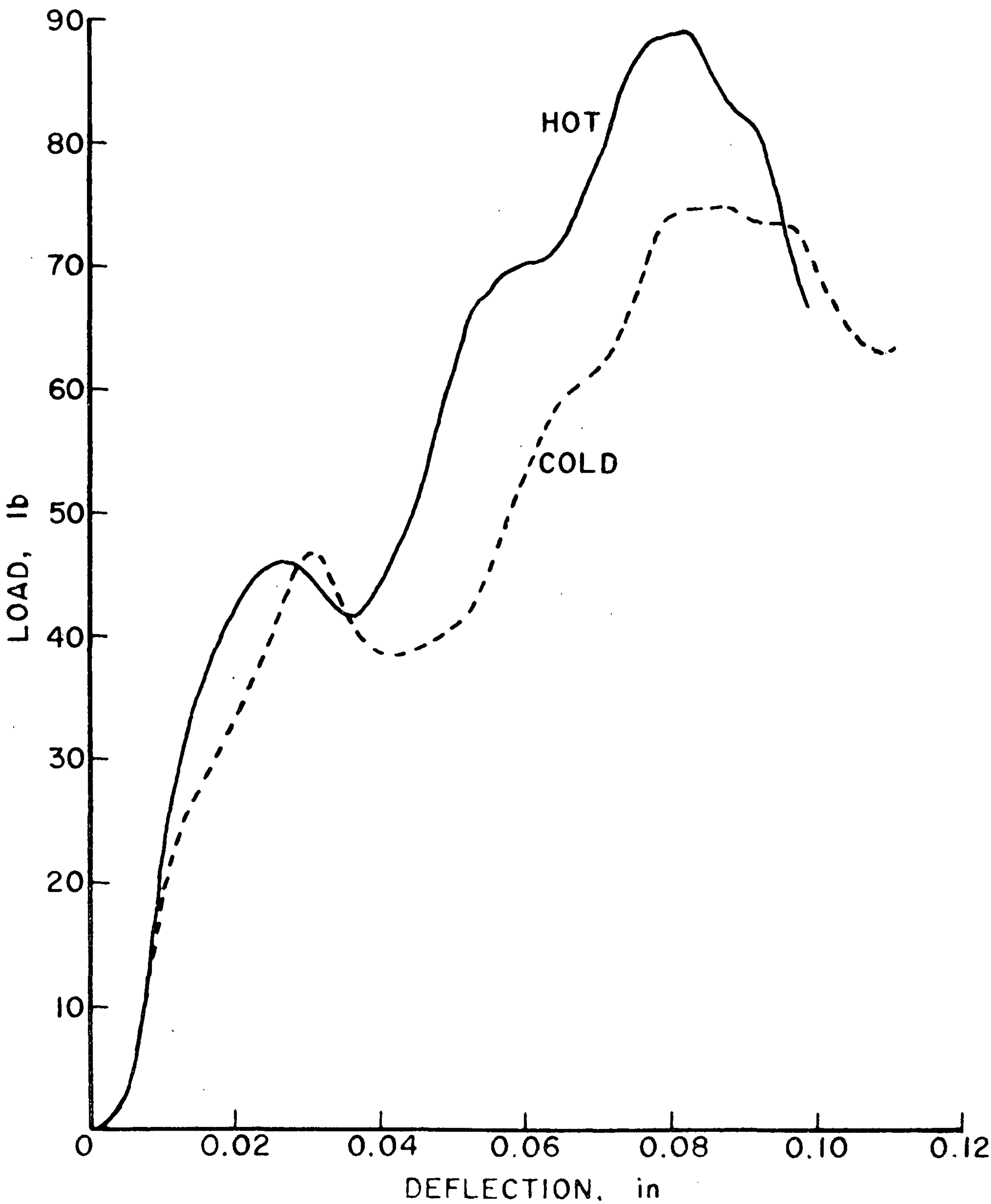

Figure 8. Typical flat crush load-deflection curves for cold and hot formed C-flute board.

stiffness and weight will have no effect on the degree of caliper reduction and degradation in other properties dependent on the effective caliper. Most of the literature discusses roller crushing effects.

On the other hand, if boards (hot or cold formed) are subjected to the same transverse stress, boards with the stiffest mediums will crush the least. Thus, for a given load, the board with the stiffer, heavier medium should exhibit less degradation in board properties. This stressing process is more likely to occur in transportation, e.g., humping operations.

Early work at the Institute was concerned with the effect of crushing on combined board properties and the flat crush fatigue behavior of combined board (hot corrugated) (22,23). The shape of the flat crush load-deformation curve was discussed (23), and it was pointed out that loads greater than the first peak caused signficant nonrecoverable deformation, i.e., degradation of the combined board. It was also shown that combined board can withstand many repeated transverse loadings if the applied loads are kept below the first peak load.

In more recent work, Staigle (24), Crisp, *et al.* (25), and Nordman, *et al.* (26) discussed the crushing of hot formed combined board in the converting process by feed rolls and belts. Staigle (24) showed that the permanent loss in caliper, as measured using the TAPPI procedure, is much less than the actual caliper reduction when board is passed between steel rolls. Thus, a permanent caliper loss of 10 mils may result from a roll crush treatment of 40 mils. In Fig. 8, a 40 mil reduction in caliper would cause nonreversible deformations, and degradation of board properties would be expected even though the permanent caliper loss is much less than 40 mils. Crisp (25) also crushed board between rollers and evaluated the changes in board properties. He defined "hardness" as the greatest flat crush load up to 0.10 inch deflection and concluded hardness was more sensitive to board damage than caliper. Thus, the early portion of the flat crush curve was considered to be more important insofar as box plant crushing is concerned, although hardness decreased much more than box compression. The flat crush test was not suitable as a damage indicator because the maximum flat crush load was not affected by small amounts of crushing.

Nordman, *et al.* (26) studied crushing of hot formed combined board between steel rolls and confirmed Staigle's work on caliper recovery. The major portion of the reduction in thickness due to roller crushing is recovered. However, the effective structural thickness is actually decreased because the fluted medium is damaged. Thus board properties dependent on the effective thickness are reduced. Both Crisp and Nordman found that flexural stiffness, caliper, first peak flat crush load, and box compression decrease with increasing degrees of crush, particularly as the flat crush first peak deformation is exceeded. However, the decreases in box compression strength do not necessarily accelerate until extreme degrees of crush are imposed. Cold formed board should exhibit the same trends as obtained in the above studies on hot formed board.

Morris (27) has emphasized that satisfactory container performance during transportation is important. In transportation, the loaded box must cope with repeated applications of stress at low to high levels. He contends that medium stiffness is an important factor in maintaining box compression performance potentials through the transportation environment. He believes the field box continues to function until we crush the legs of the flute, i.e., flat crush test failure. This contention is probably consistent with the box results after crushing which were obtained by Crisp and Nordman (25,26). As a final comment, the results in Ref. (26) show that precrushing board to given stress levels causes reductions in box compression which vary with medium stiffness as well as flute caliper and other factors. To summarize flat crush performance requirements,

(1) The initial portion of the flat crush load-deflection curve is more critical than ultimate flat crush in assessing whether a given degree of crushing will degrade board quality. In this regard, cold formed flutes should perform as well as hot formed flutes.

(2) The entire load-deflection curve is important to box performance because boxes can continue to function even though the crushing has exceeded the first peak deflection. For this aspect of end-use performance, cold formed board may show a slight disadvantage. One of the aims of this research was to improve corrugating medium with respect to flat crush performance. Results from this work should be applicable to both processes.

2. Strength Losses in Fluting

For our initial work, we focused attention on the machine direction (m.d.) and cross direction (c.d.) edgewise compressive characteristics of the medium as related to forming conditions. The compressive characteristics of the medium affect the converting performance of combined board and end-use box performance. The medium contributes directly to c.d. short column strength (along with the liners) and indirectly to flexural stiffness, where it serves primarily to maintain the desired liner separation. The latter involves the flat crush load-deformation characteristics of the formed medium and, hence, the m.d. edgewise compressive properties of the medium. Crushed combined board, whether in converting or end-use, is a weak product. Thus the compressive characteristics of the medium in both directions are involved in box performance.

Accordingly, we planned and carried out work in the following area:

1. Evaluation of the effect of cold forming conditions on the compressive characteristics of the medium and comparison with hot formed medium. Other properties such as tensile and bonding strength were also considered.
2. Effect of forming conditions on flute shape.
3. Relation of flat crush to medium properties - structural models.

Most of our work was carried out using four commercial 26-lb mediums. There were three semichemical mediums and one recycled fiber medium. As expected, the recycled fiber medium contained a higher percentage of long softwood fiber than the semichemical mediums. Two of the mediums exhibited about equal flat crush under cold and hot corrugating conditions; the other two mediums exhibited lower cold than hot flat crush. The physical characteristics of the mediums are summarized in Section E together with the general procedures employed.

To determine the degree of change in the edgewise compressive characteristics of formed medium, we made short span compressive tests on fluted but unbonded sections of cold and hot formed medium. The compressive tests were made on the STFI strip compressive tester which employs a test span of 0.7 mm (28). The short test span permits localized strength determinations which are of great value in studying formed flutes.

The machine direction STFI compressive results taken at various positions around the flute are shown in Figs. 9 and 10 for the "different" and "equal" cold/

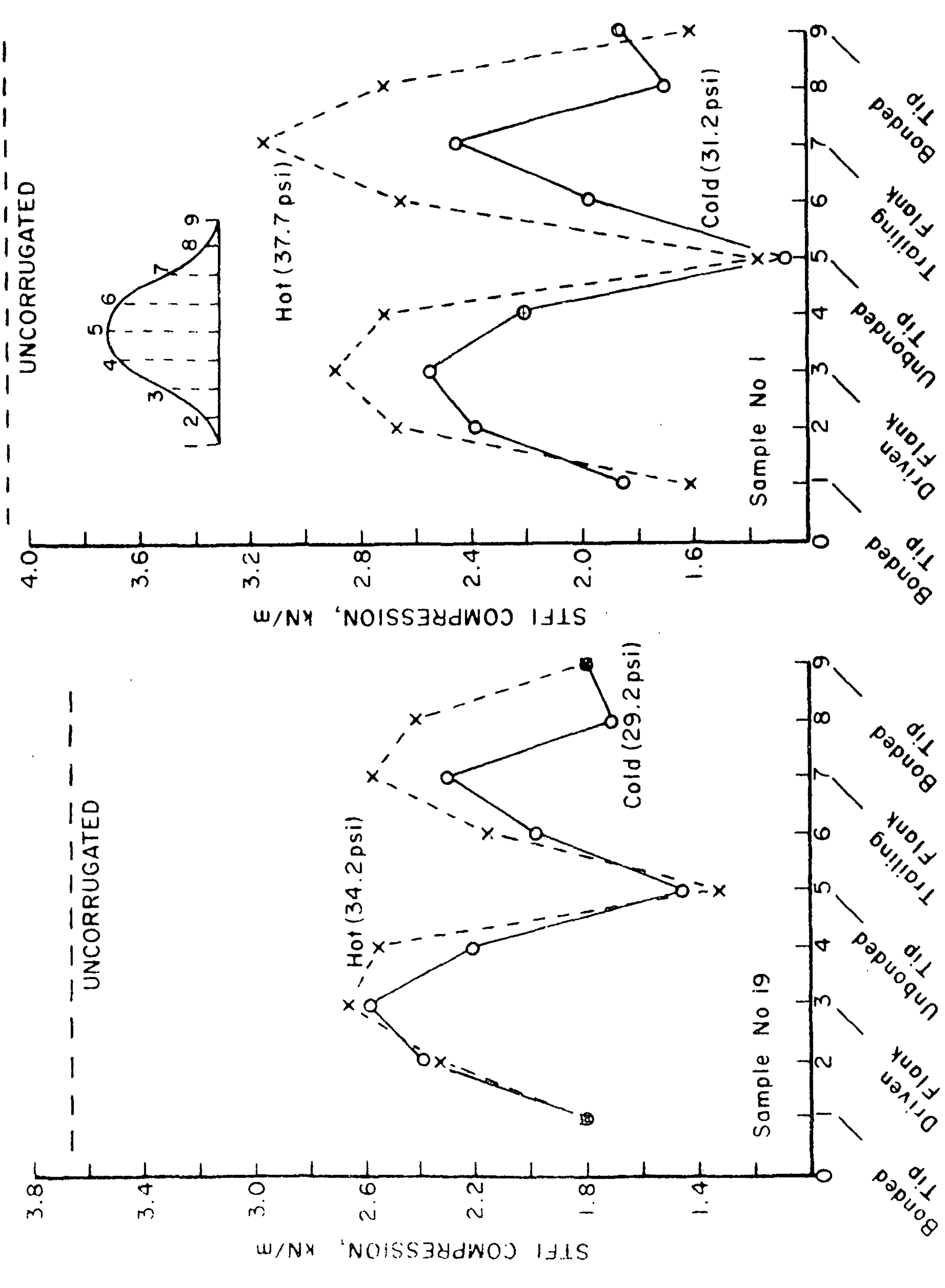

Figure 9. Machine direction compressive strength after fluting for mediums exhibiting "different" cold/hot flat crush strength (flat crush values in parentheses).

hot flat crush mediums, respectively. The results show that the formed medium exhibits much lower compressive strengths than the uncorrugated medium. This is true for both hot and cold formed medium, although there are some significant differences which are noted below. Overall, the reductions in m.d. compressive strength were about 42%. We believe the reductions in compressive strength reflect fiber bonding damage caused by the high stresses in the forming process.

The m.d. compressive strengths in the flank and tip/flank regions (positions 2-4 and 6-8) tended to be somewhat lower on the cold formed medium than the hot formed medium in Fig. 9. This was more evident on the trailing flank. As mentioned, the two mediums in Fig. 9 exhibited lower cold than hot flat crush. In the case of the mediums where the cold and hot flat crush results were comparable, the compressive strengths of the hot and cold formed mediums were also about the same (Fig. 10).

Figure 11 shows that the STFI compressive strengths in the tip-flank region are well related to flat crush for both the hot and cold formed boards. In contrast, the STFI compressive results on the uncorrugated medium were not well related to the flat crush results on the cold-formed medium. This helps confirm that degradation of the m.d. edgewise compressive potentials of the medium due to forming is a factor in flat crush performance.

When single-faced board is tested in flat crush, the unbonded flute tip flattens and squares off as the first load peak is reached (see Fig. 12). Note that the cold formed board deformed less symmetrically and exhibits a broader flattened off portion. This would be expected if the compressive strength of the trailing flank is less than that of the leading flank, as illustrated in

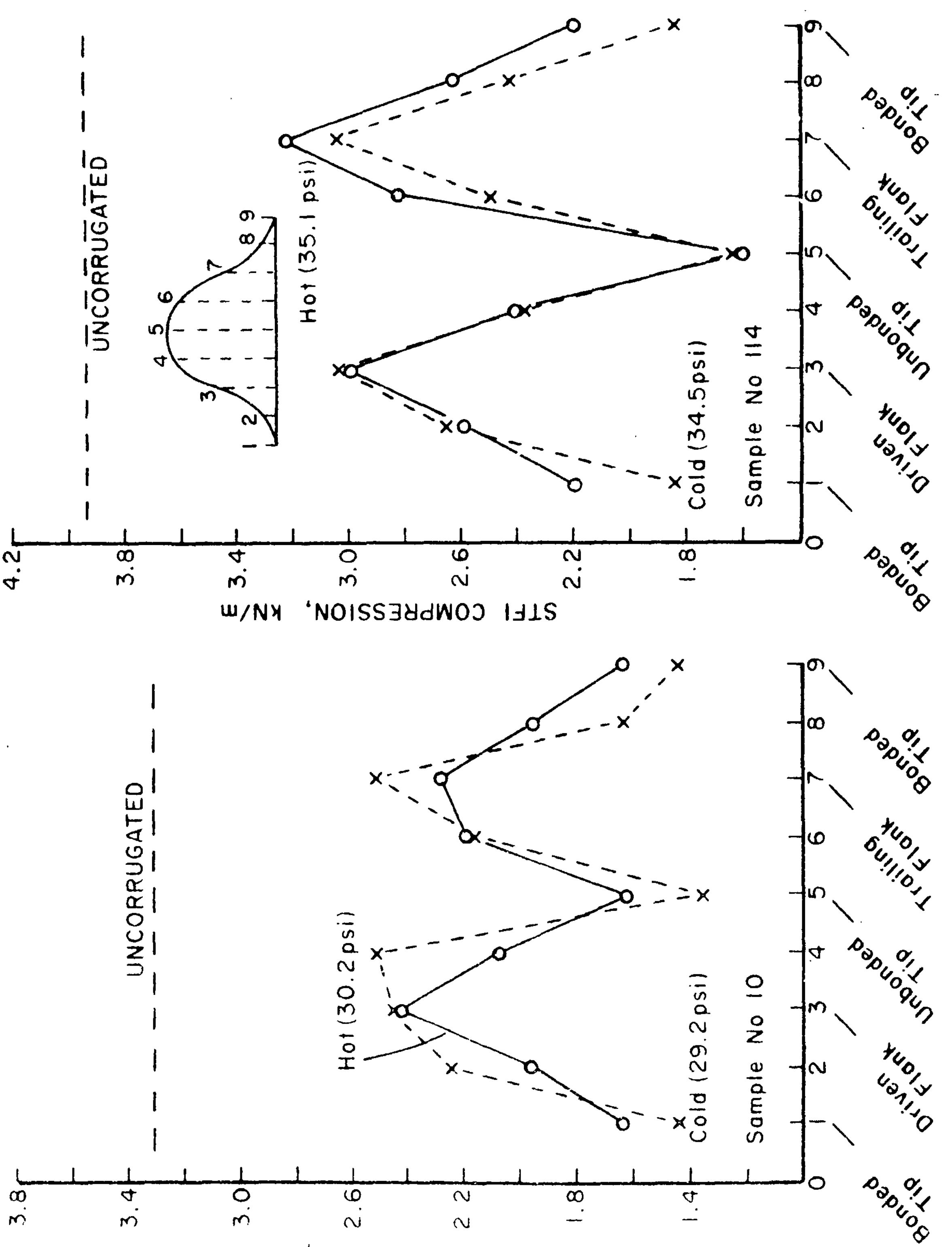

Figure 10. Machine direction compressive strength after fluting for mediums exhibiting "equal" cold/hot flat crush ratios (flat crush values in parentheses).

hot flat crush mediums, respectively. The results show that the formed medium exhibits much lower compressive strengths than the uncorrugated medium. This is true for both hot and cold formed medium, although there are some significant differences which are noted below. Overall, the reductions in m.d. compressive strength were about 42%. We believe the reductions in compressive strength reflect fiber bonding damage caused by the high stresses in the forming process.

The m.d. compressive strengths in the flank and tip/flank regions (positions 2-4 and 6-8) tended to be somewhat lower on the cold formed medium than the hot formed medium in Fig. 9. This was more evident on the trailing flank. As mentioned, the two mediums in Fig. 9 exhibited lower cold than hot flat crush. In the case of the mediums where the cold and hot flat crush results were comparable, the compressive strengths of the hot and cold formed mediums were also about the same (Fig. 10).

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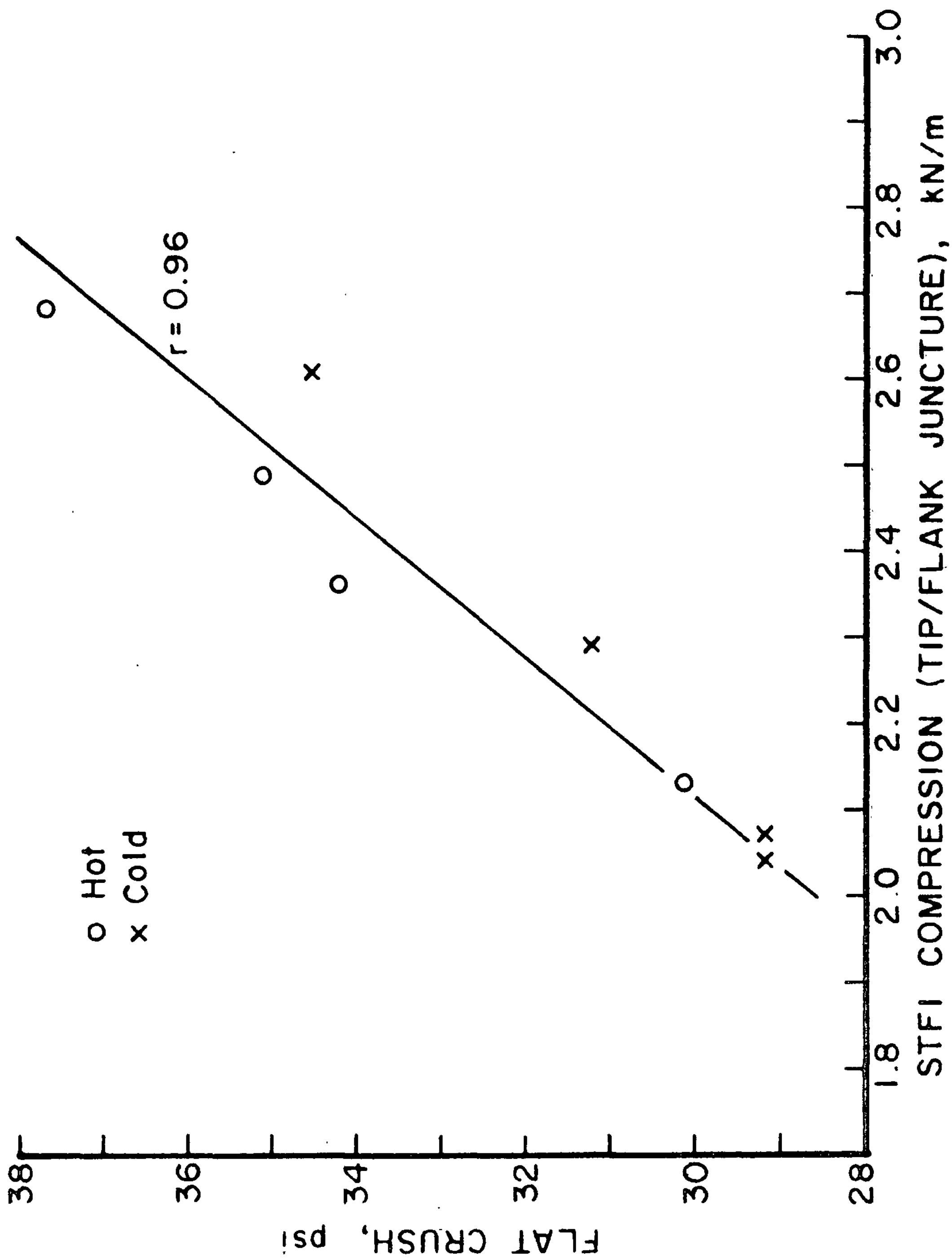

Figure 11. Relation between compressive strength of the fluted medium and flat crush.

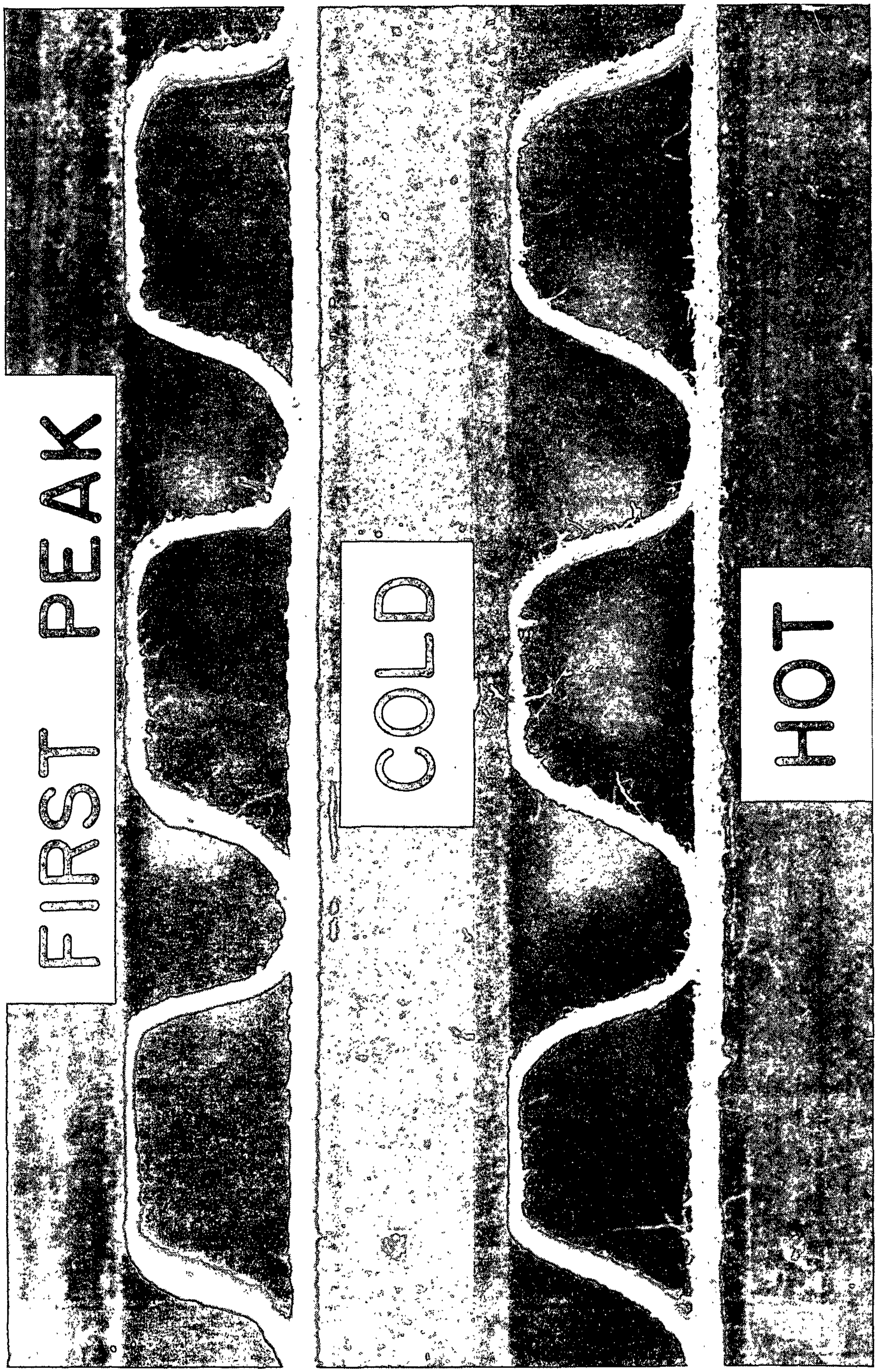

Figure 12. Appearance of cold and hot formed board at flat crush failure (trailing flank to right).

Fig. 9. The flank/tip regions are particularly critical because these are the "hinge" points in the flat crush test structure.

At failure in flat crush the medium forms a hat shaped (frame) structure (see Fig. 13). The figure also illustrates that the cold-formed medium failed in a less symmetrical manner than the hot medium. In this example, final failure appeared to be associated with the trailing flank for both forming conditions.

Based on observation and experiment, we believe it is reasonable to expect that the flat crush load-deformation characteristics should be related to the m.d. edgewise compressive properties of the formed medium. Thus, the lower ultimate flat crush strength obtained with some mediums under cold conditions is a result of the greater compressive strength degradation in the flank/tip and flank regions of the flute.

Short span m.d. tensile tests on the formed medium showed reductions in strength ranging from about 17-44% compared to the uncorrugated medium on sample 1 (Table XIII and Fig. 14). In all cases, the percentage reductions in tensile strength were less than in edgewise compressive strength, often much less. Thus, while forming affected both the tensile and compressive characteristics of the medium, compressive strength was more drastically lowered. We speculate this occurs because compressive strength is more sensitive to the delamination stresses which are induced in the forming process.

Figure 14 shows that the cold formed medium generally exhibits lower tensile strength than hot formed medium in all flute positions. However, the tensile strengths varied more erratically with flute position than compressive strength. In any case the reductions in tensile strength did not appear to be

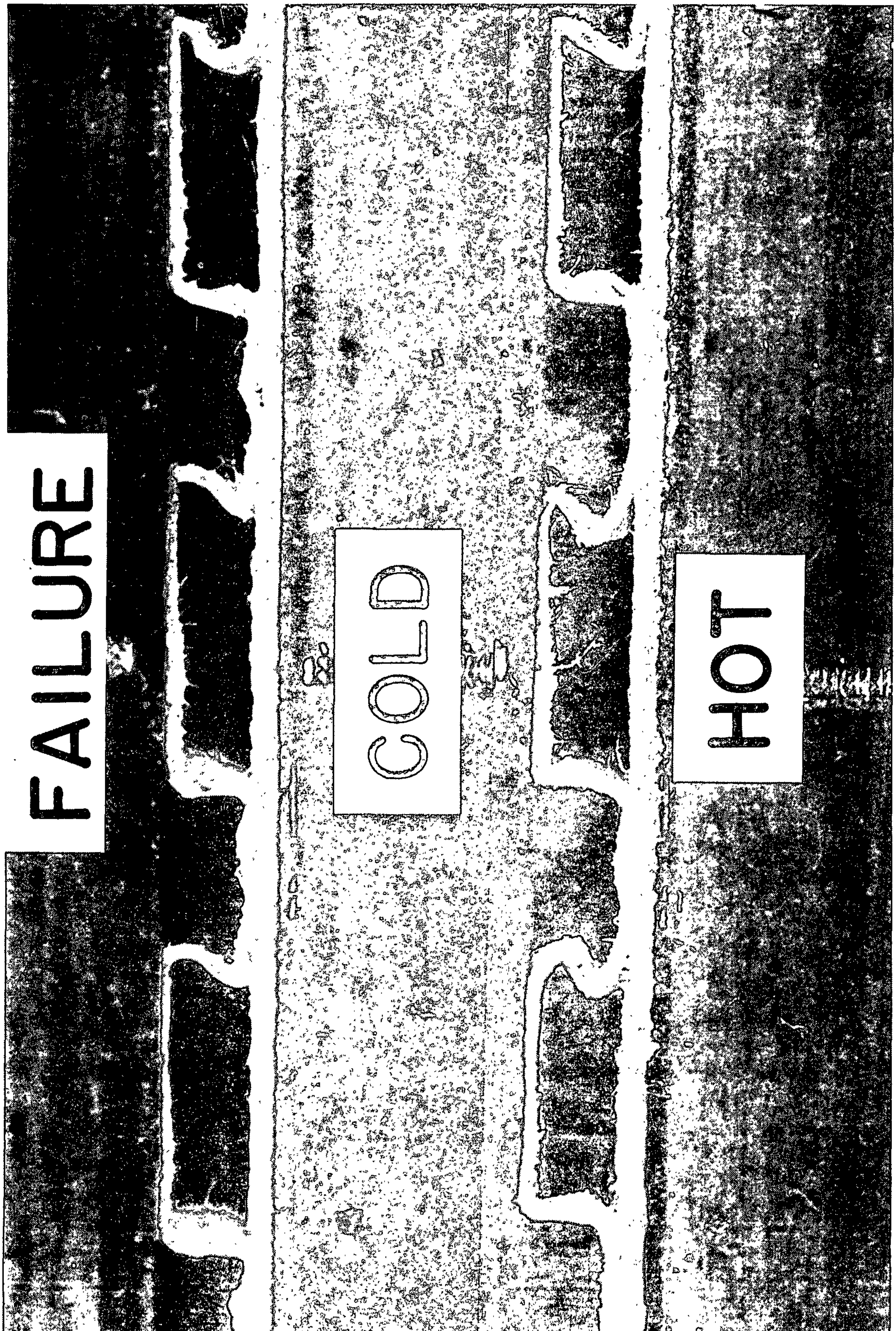

Figure 13. Appearance of cold and hot formed board at flat crush failure (trailing flank to right).

TABLE XIII

EFFECT OF FORMING ON THE LOCAL TENSILE AND COMPRESSIVE STRENGTH OF THE MEDIUM

(Medium Sample 1)

Flute Position	Forming Condition	Compressive Strength,[b] kN/m	Diff., %[a]	Short Span Tensile,[b] kN/m	Diff., %[a]
Uncorrugated	--	4.8	--	10.7	--
Corrugated					
Bonded root	Cold	1.86	-54.4	7.32	-31.6
	Hot	1.61	-60.5	7.65	-28.5
Root/driven flank	Cold	2.40	-41.2	6.58	-38.5
	Hot	2.67	-34.6	8.89	-16.9
Drive flank	Cold	2.55	-37.5	8.25	-22.9
	Hot	2.89	-29.2	7.62	-28.8
Unbonded tip/ driven flank	Cold	2.32	-43.1	6.63	-38.0
	Hot	2.71	-33.6	7.37	-31.1
Unbonded tip	Cold	1.28	-68.6	6.01	-43.8
	Hot	1.37	-66.4	8.65	-19.2
Unbonded tip/ trailing flank	Cold	2.11	-48.3	6.04	-43.6
	Hot	2.65	-35.0	8.35	-22.0
Trailing flank	Cold	2.45	-40.0	8.09	-24.4
	Hot	3.14	-23.0	8.05	-24.8
Bonded root/ trailing flank	Cold	1.86	-54.4	7.93	-25.9
	Hot	2.70	-33.8	8.30	-22.4

[a]Based on uncorrugated results as reference.
[b]STFI compressive and short span tensile spans were 0.7 and 1.27 mm, respectively.

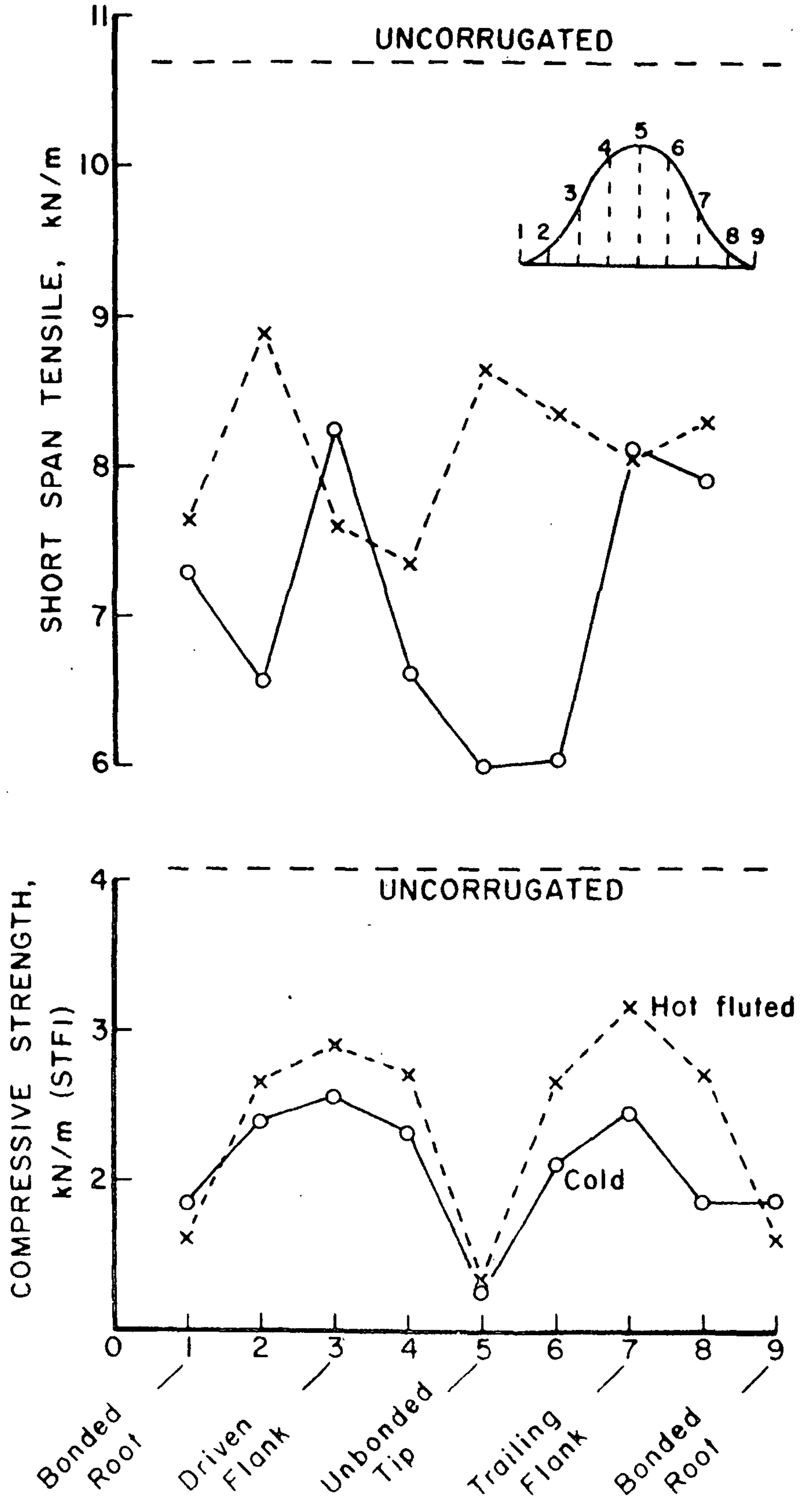

Figure 14. Effect of forming on short span tensile and edgewise compressive strength of Sample 1.

directly related to the cold-hot flat crush differences, although they may affect other board qualities. This is not surprising because the flat crush load-deformation characteristics would be expected to be primarily dependent on the compressive characteristics of the medium.

We also carried out STFI edgewise compressive tests in the cross-machine direction on hot and cold formed medium. No separation by position on the flute was possible. Figure 15 indicates that forming reduces the edgewise compressive strength potentials of the medium in the cross direction. This is probably due to delamination induced by shear stresses in forming as mentioned previously. The reductions are in the neighborhood of 20 to 30% and are not greatly different for hot and cold corrugating. Top load box compressive strength is dependent, in part, on the cross direction strength of the medium. Thus, these findings are significant because they indicate that the corrugating process degrades the compressive potentials of the medium in the c.d. as well as in the m.d.

In addition to determining how forming affects compressive strength, tests of bonding strength and the transverse shear characteristics of the medium were carried out. In Fig. 16 the Viscosity-Velocity Product (VVP) type bonding strengths in the machine direction of the hot and cold formed mediums are significantly lower than for the uncorrugated medium. Cold forming tends to give slightly greater reductions. It appears likely that the reductions in edgewise compressive strength are due to these losses in bonding strength. This seems particularly true in view of the delamination which accompanies compressive failure. Losses in shear strength may also be involved and work is in progress to develop a transverse shear test. We should note that the bonding strength tests on the formed medium involve nonideal test conditions; therefore the

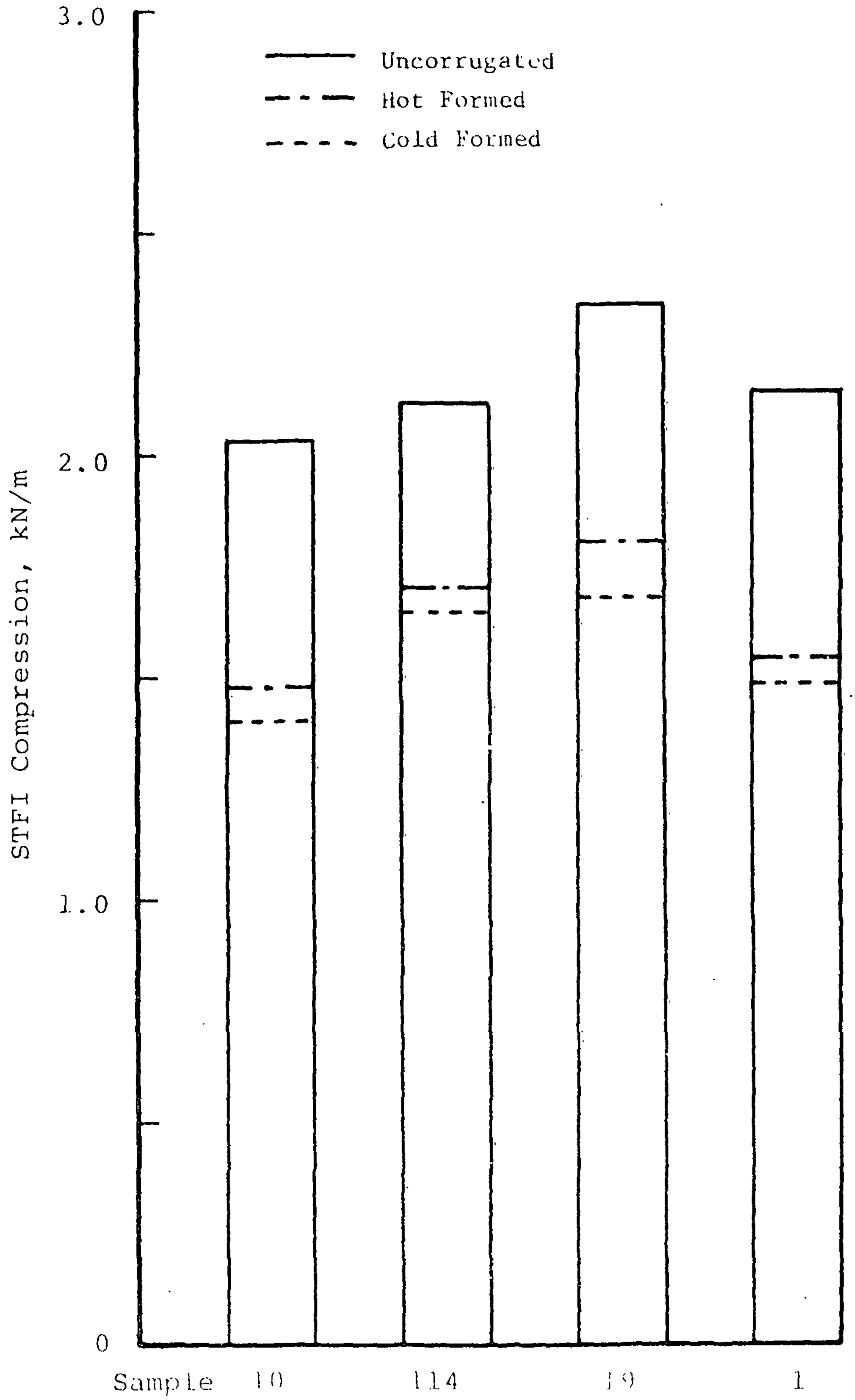

Figure 15. Effect of forming on cross-direction edgewise compressive strength.

results are probably only an approximation to the state of the medium after forming.

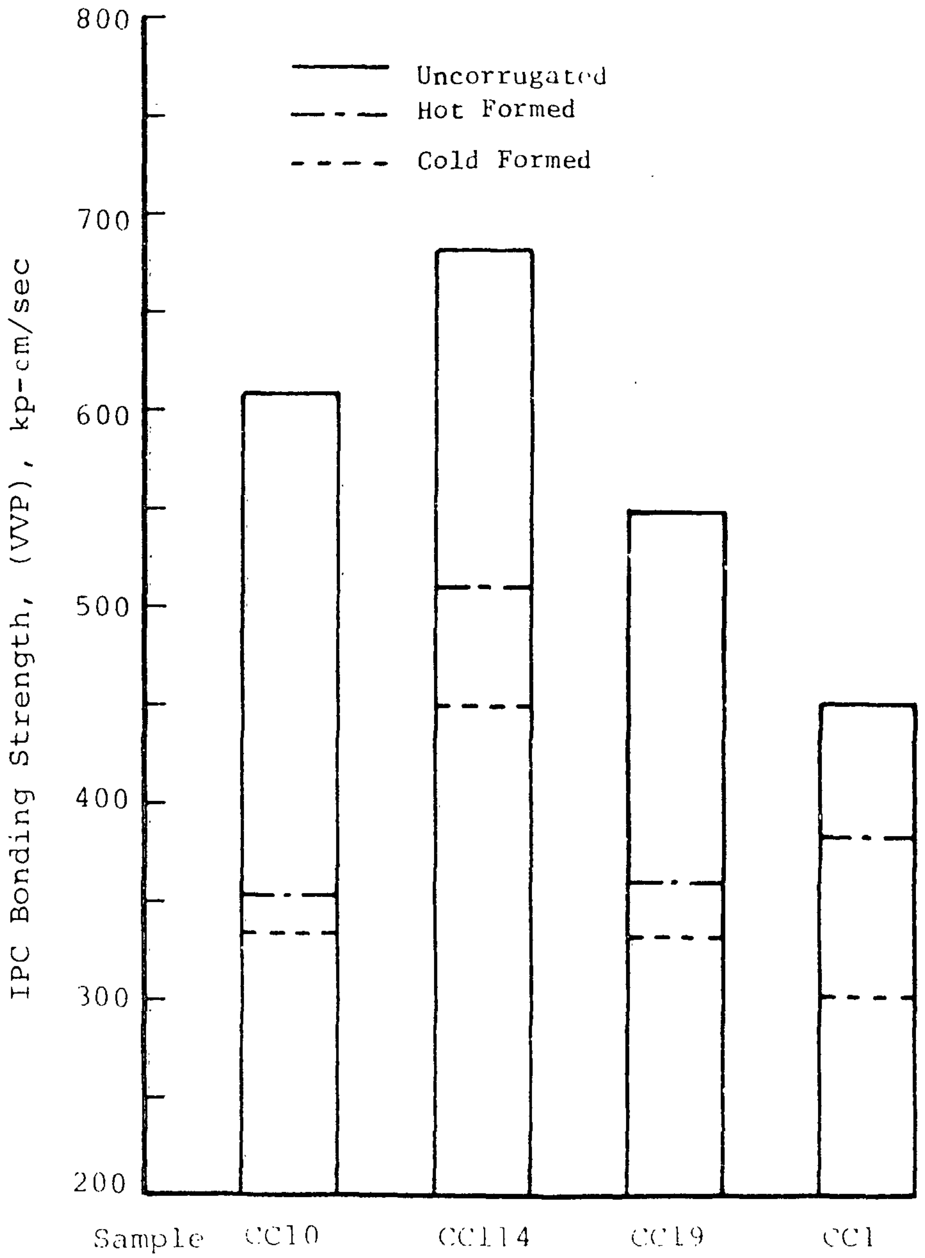

Figure 16. Effect of forming on transverse bonding strength in the machine direction.

Thickness direction tensile (ZDT) bonding strength tests were also carried out as shown in Fig. 17. Generally, the formed mediums exhibited lower ZDT strengths than the uncorrugated medium. However, the decreases in ZDT strength varied considerably from medium to medium and the decreases were probably not significant in some cases. These results appeared to be less revealing than the VVP type tests.

From these results it appears that

1. The edgewise compressive and tensile strengths of medium are greatly reduced by fluting under both hot and cold conditions. This is probably due to fiber-fiber bond damage during fluting.
2. Some mediums show more evidence of compressive strength reduction under cold conditions than under hot conditions. We believe this accounts for the lower flat crush obtained with such cold formed mediums.
3. We also noted that some mediums tend to exhibit more compressive degradation on the trailing flank than on the driven flank under cold fluting as compared to hot fluting conditions.

3. Strength Loss-Forming Stress Relationships

Experiments to determine how various types of corrugating stresses bring about compressive strength reductions were conducted. Our initial experiments involved prestressing a medium sample in tension, and combined bending and tension at standard test conditions, 73°F and 50% RH. The properties of the medium under these test conditions will approximately correspond to those in cold corrugating.

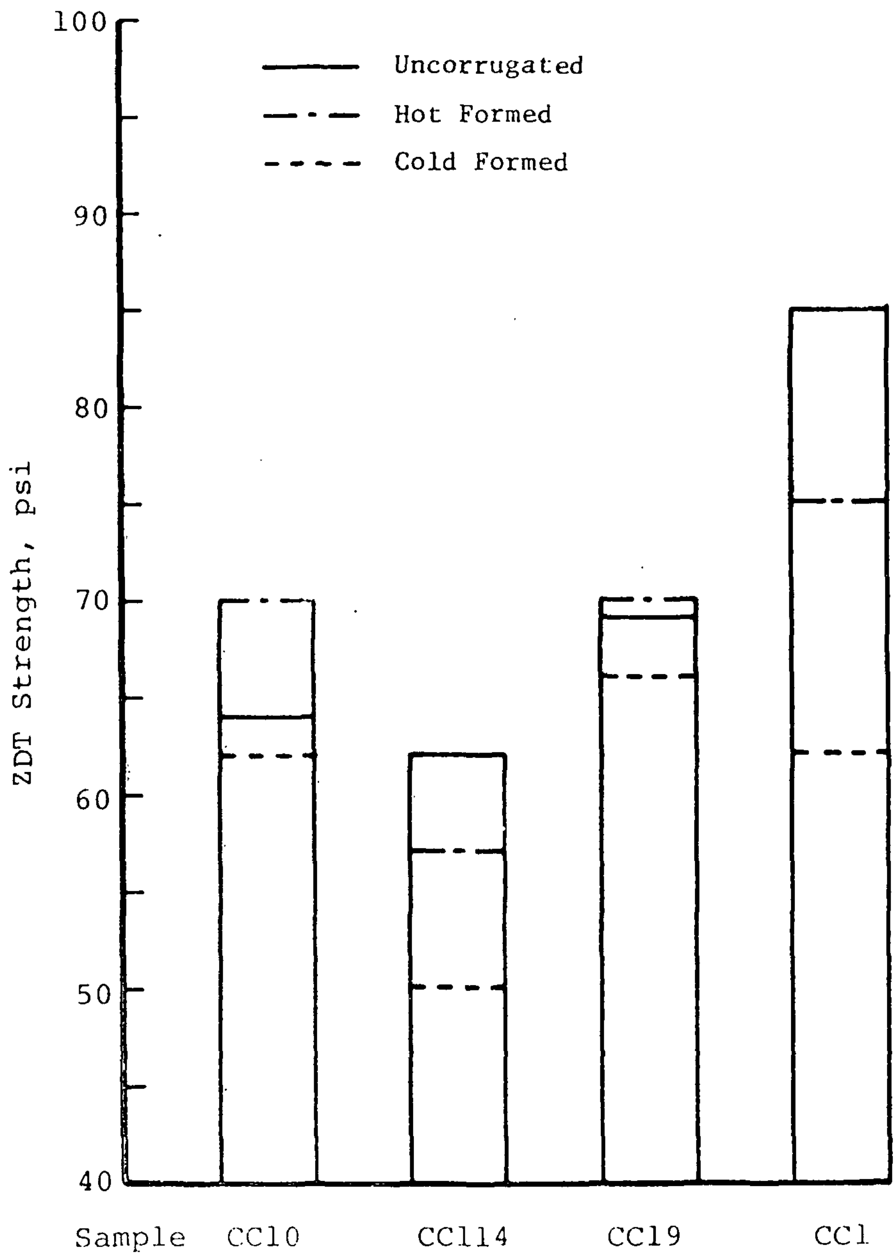

Figure 17. Effect of forming conditions on ZDT strength.

As a first step, we loaded m.d. specimens of medium in tension to failure. The remnants were then evaluated for compressive strength using the STFI tester. Figure 18 shows that prestressing in tension has little or no effect on compressive strength.

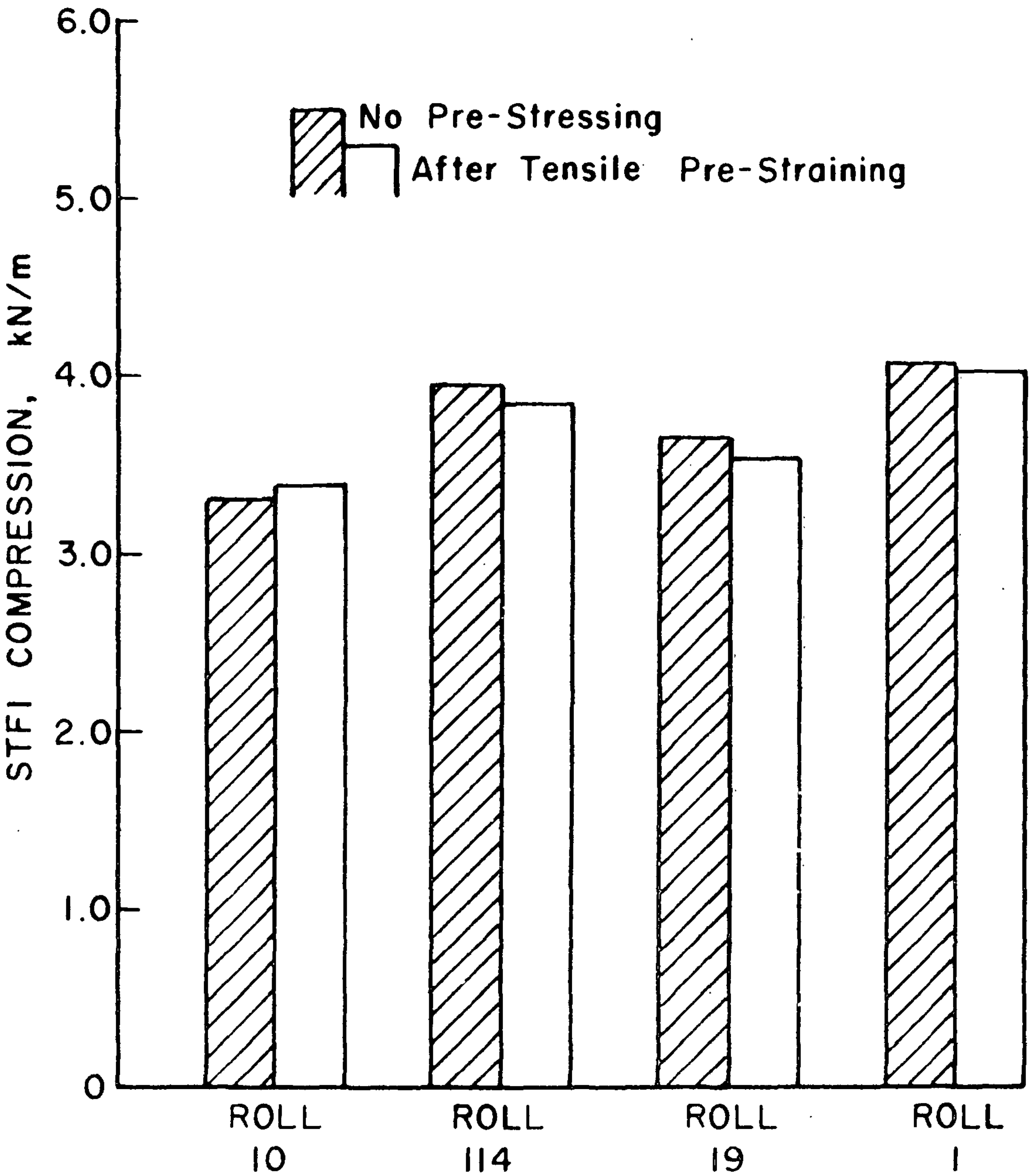

Figure 18. Effect of tensile prestressing on m.d. edgewise compressive strength.

In cold or hot corrugating the onset of fracturing is usually gradual over a range of speeds. Also, fractures do not usually propagate across the web "instantaneously" as occurs in long-span tensile tests. Within the labyrinth, it is likely that the medium is sufficiently stretched to produce local fiber bond damage and to affect compressive strength, but there is insufficient stored energy to cause a tensile fracture to propagate. This behavior would be somewhat analogous to a short span tensile test on a "stiff" tester. In such tests there is often no visible indication of failure at the maximum load and no "instantaneous" failure is encountered. However, compressive strength might be lowered under such conditions.

When medium is preflexed by bending it around a small radius under low tension, the m.d. compressive strength after flexing is greatly reduced (Fig. 19). The smaller the radius, the greater the loss in compressive strength. These results show that the bending stresses imposed during forming could cause the losses in m.d. compressive strength of the fluted medium. Because the tip and root radii of the corrugating rolls are relatively small, both bending and shear stresses must be large for the medium to conform to the contour, as mentioned earlier.

The combined effects of bending and tension are illustrated in Fig. 20. Figure 20 shows that the compressive strength decreases rapidly as the wrap angle used in flexing increases from 0 to about 90°. Contact angles between the medium and the flute tip are about 90-120° near the center of the corrugating labyrinth (4,5). The results in Fig. 20 also indicate that the losses in compressive strength are aggravated by higher tensions and smaller radii. Past work has indicated that high web tensions occur in the corrugating labyrinth as friction between the medium and steel rolls increases. We believe that Fig. 20 indicates that high web tensions adversely affect the properties of the formed board such as compressive strength.

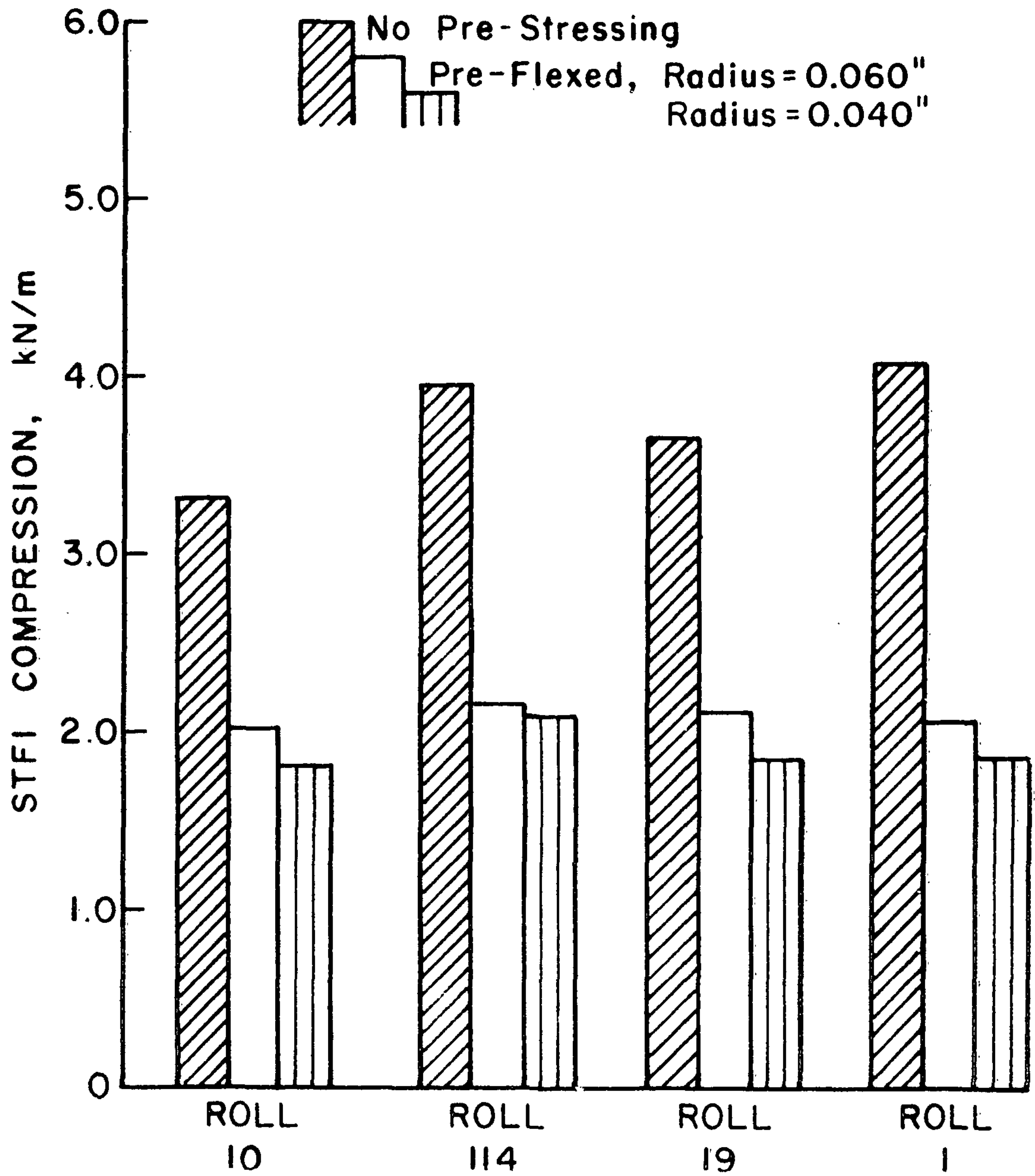

Figure 19. Effect of bending prestressing on m.d. compressive strength.

The moisture content of the medium at time of forming will affect stiffness and moldability. Under cold forming conditions, higher moisture contents should permit the medium to be bent to the flute radius with less damage and enhance molding for flute shape retention. This assumes that friction is held constant or reduced. We have partially confirmed this in past cold corrugating trials over a limited moisture range, indicating that higher moisture content promotes higher flat crush.

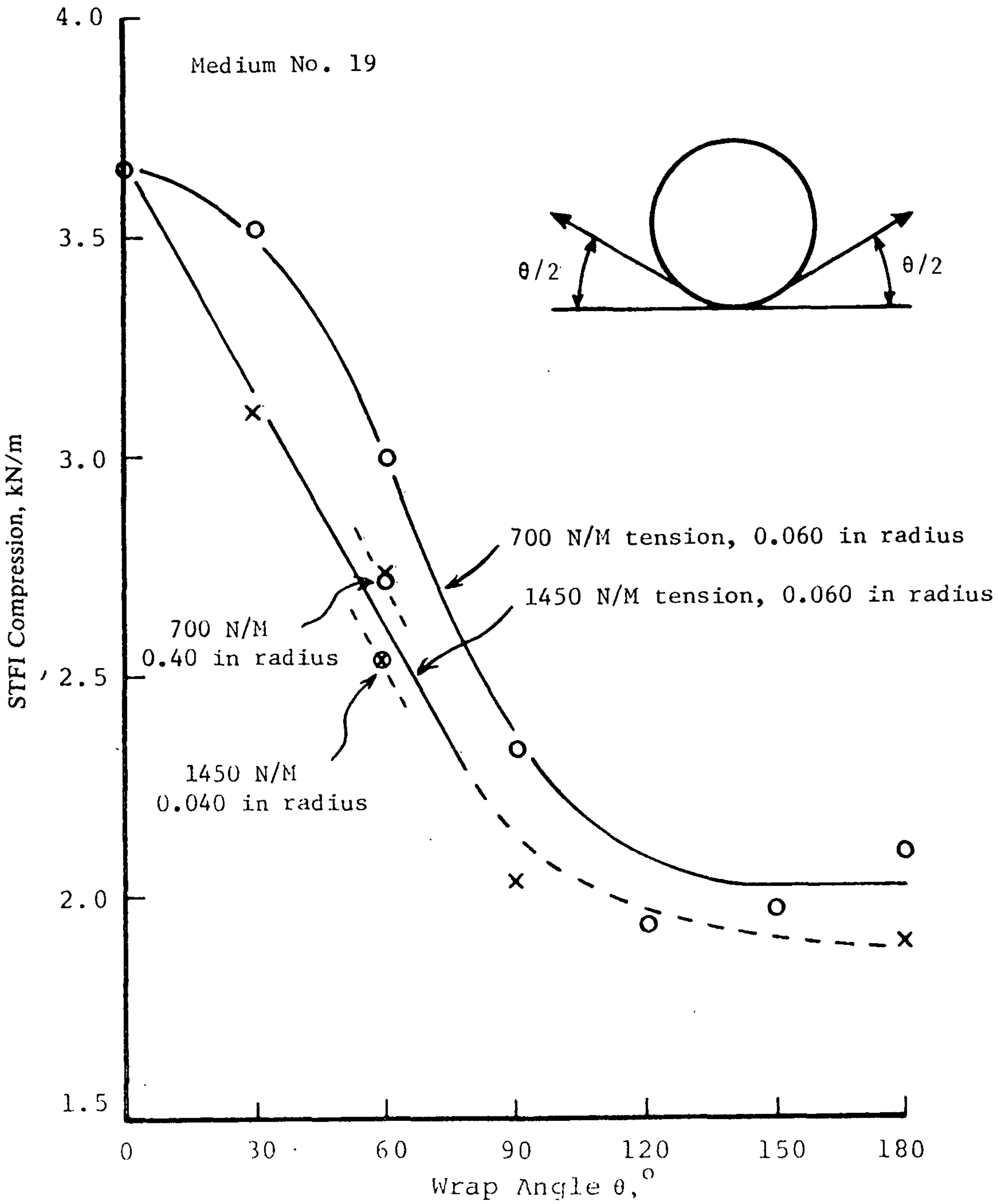

Figure 20. Effects of tension and flexing conditions on compressive strength.

To determine how flexing at various moisture contents would affect compressive strength, trials were carried out at RH levels ranging from about 15% to 90%. Figure 21 shows that the compressive strengths of the flexed mediums decreased at about the same rate as the unstressed control with increased levels of moisture.

Limited trials were also carried out in which the medium was preflexed at various moisture contents and then reconditioned to 50% RH prior to compression testing. It was expected that the flexing at high RH would have less severe effects than at 50% RH and, hence, would not reduce compressive strength as much. However, the effects of the high RH flexing seemed to result in about the same compressive strengths at 50% RH as flexing and testing at 50% RH. We did note, however, that the reductions in compressive strength for the various mediums seemed to be related to the VVP bonding strength.

4. Shearing Stress and Clearance Factors

In the forming process (cold or hot) large bending and shear stresses are induced in the medium as it is formed to the flute contour. A qualitative analysis shows that formation of the flute cannot occur by pure bending because this would require very large machine direction stretch values (3-5).

The results of a qualitative analysis are illustrated in Fig. 22. If the medium assumes the flute shape by bending only (no shear strain), the required machine direction strain is at least 8%. Thomas (5) indicates even higher stretch values may be necessary. On the other hand, if only shear stresses were allowed, a very high shear angle would be required and the medium would have to behave like a deck of cards. The apportionment of the strain between bending and shear will depend on the ratio of the bending and shear stiffnesses (elastic and inelastic)

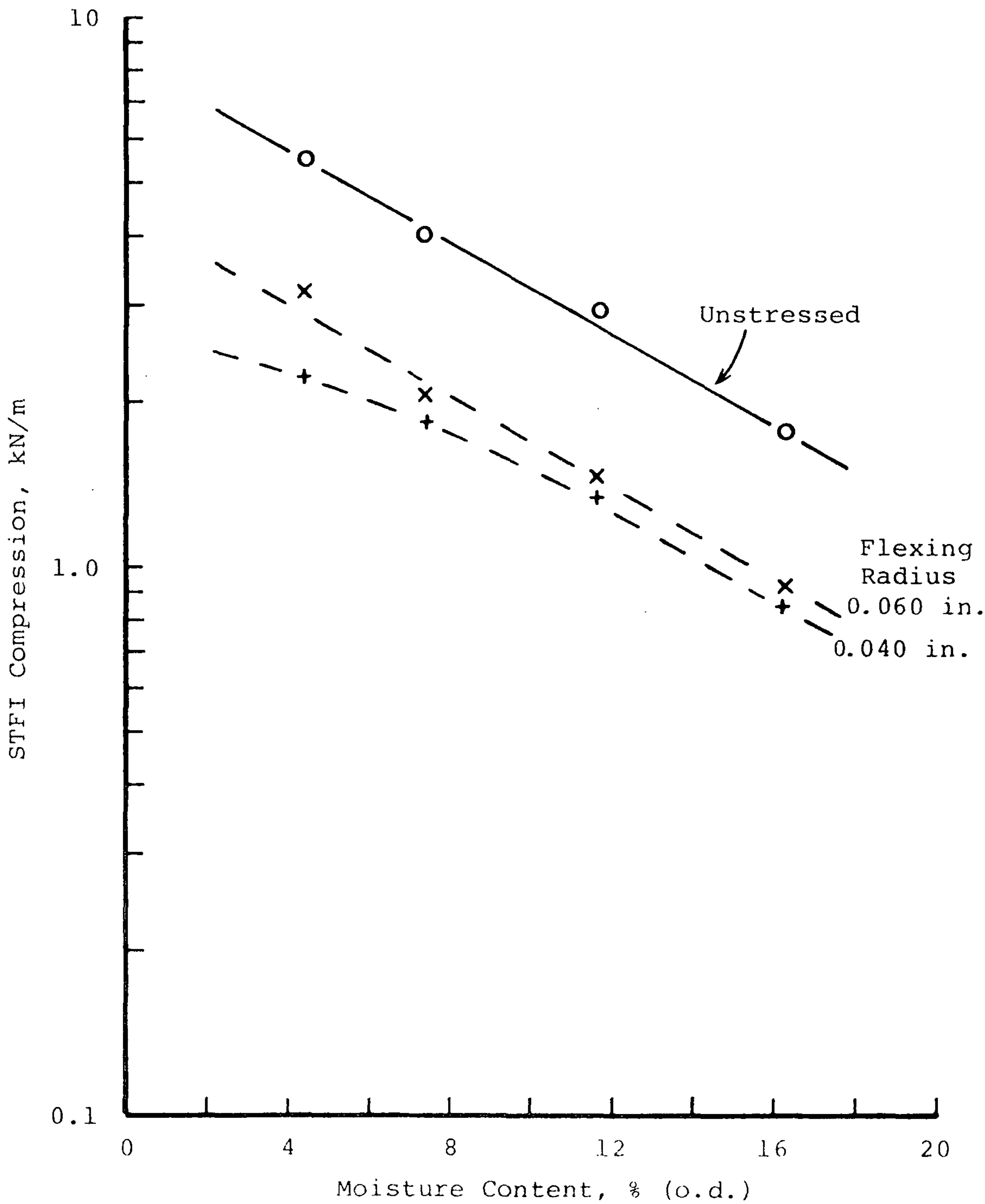

Figure 21. Effects of flexing at various moisture levels on compressive strength.

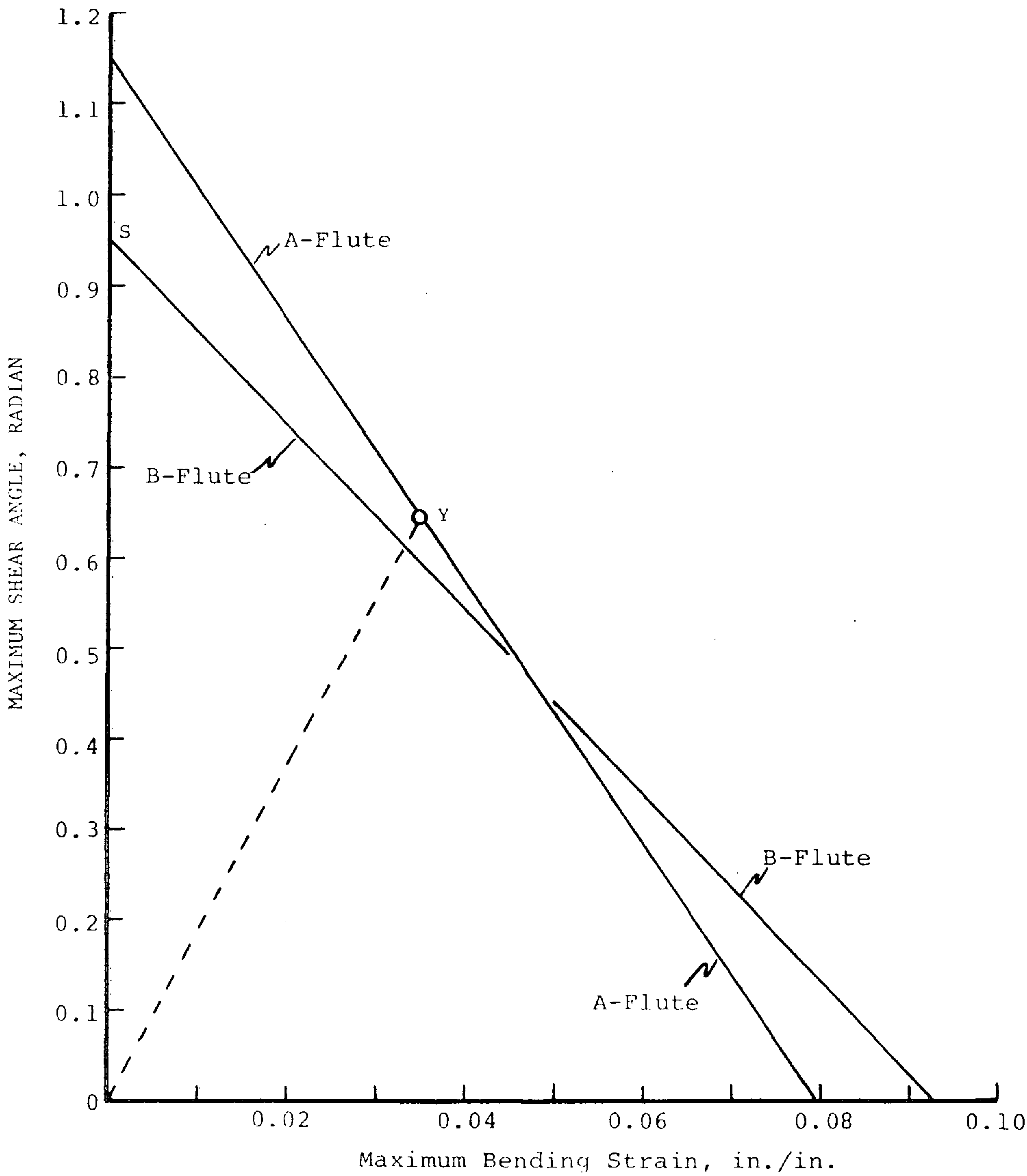

Figure 22. Relationship between bending and shear strain required for flute forming [Ref. (9)].

of the medium. If the medium is stiffer in bending than in shear, flute formation will involve large shear strain and small bending strain, and vice versa. As an example, for given shear and bending properties, the strains will increase along some path OY in Fig. 22 as the flute is formed. The intersection point shows what strain levels the medium must withstand. In this example, the medium would have to have an available stretch in excess of 3.5% and an allowable shear angle in excess of 0.65 radian to form without failure. Thus we believe that shear properties of the medium are important in corrugating as in folding operations.

Preliminary ultrasonic measurements of transverse shear and extensional moduli for the four commercial test mediums are shown in Table XIV. The m.d. tensile modulus E_x is 30-40 times greater than the transverse shear modulus G_{xz} at 73°F and 50% RH. The low shear moduli indicate that a portion of the molding will depend on shear. These test results are applicable to cold forming; however, we expect similar differences between in-plane and shear moduli under hot forming conditions.

TABLE XIV

ULTRASONIC SHEAR AND IN-PLANE MODULI

	Medium Sample Number			
	10	114	19	1
MD in-plane modulus, (E_x), psi	588,000	804,000	645,000	929,000
Shear modulus G_{xz}, psi	19,300	19,000	18,400	24,200
Ratio: E_x/G_{xz}	30.4	42.2	35.0	38.5

Transverse shear measurements are difficult to carry out. Only a few techniques are mentioned in the literature. We are attempting to develop a technique for obtaining shear load-deformation curves. These will supplement and extend the ultrasonic measurements which are restricted to "elastic" displacements. Initial results indicate that the transverse shear moduli measured mechanically will be lower than those measured ultrasonically due to strain rate effects. However, considerable work is required to improve the technique so as to better characterize material performance in forming and structural performance applications.

An analysis of clearance in the labyrinth shows that a potential pinch point exists about 1/2 flute ahead of the center line (see Fig. 23). In past work at the Institute, high speed motion photography showed that fracturing under hot conditions occurs before the medium reaches the center line, also by about 1/2 flute. This is also believed to occur in cold forming. The lower roll begins to drive the upper roll at a location about 1/2 flute ahead of the center line. If the full amount of medium has not been drawn into the last half flute, relatively high tensile strains are imposed due to the pinching action. This would result in the risk of greater medium damage and the occurrence of fracture.

5. Flute Geometry and Forming Conditions

In general, the differences in flat crush between cold and hot formed board would be expected to depend on medium characteristics and/or flute geometry. Our research has been directed to determining whether geometry or material differences due to forming conditions are more important in affecting board quality. The preceding work showed that some cold formed mediums exhibited lower compressive strength after fluting than hot formed board. In these cases the cold board also gave lower flat crush than hot board. To determine if flute profile (geometry) differences are also obtained under hot and cold corrugating conditions, a detailed analysis of flute profiles was carried out.

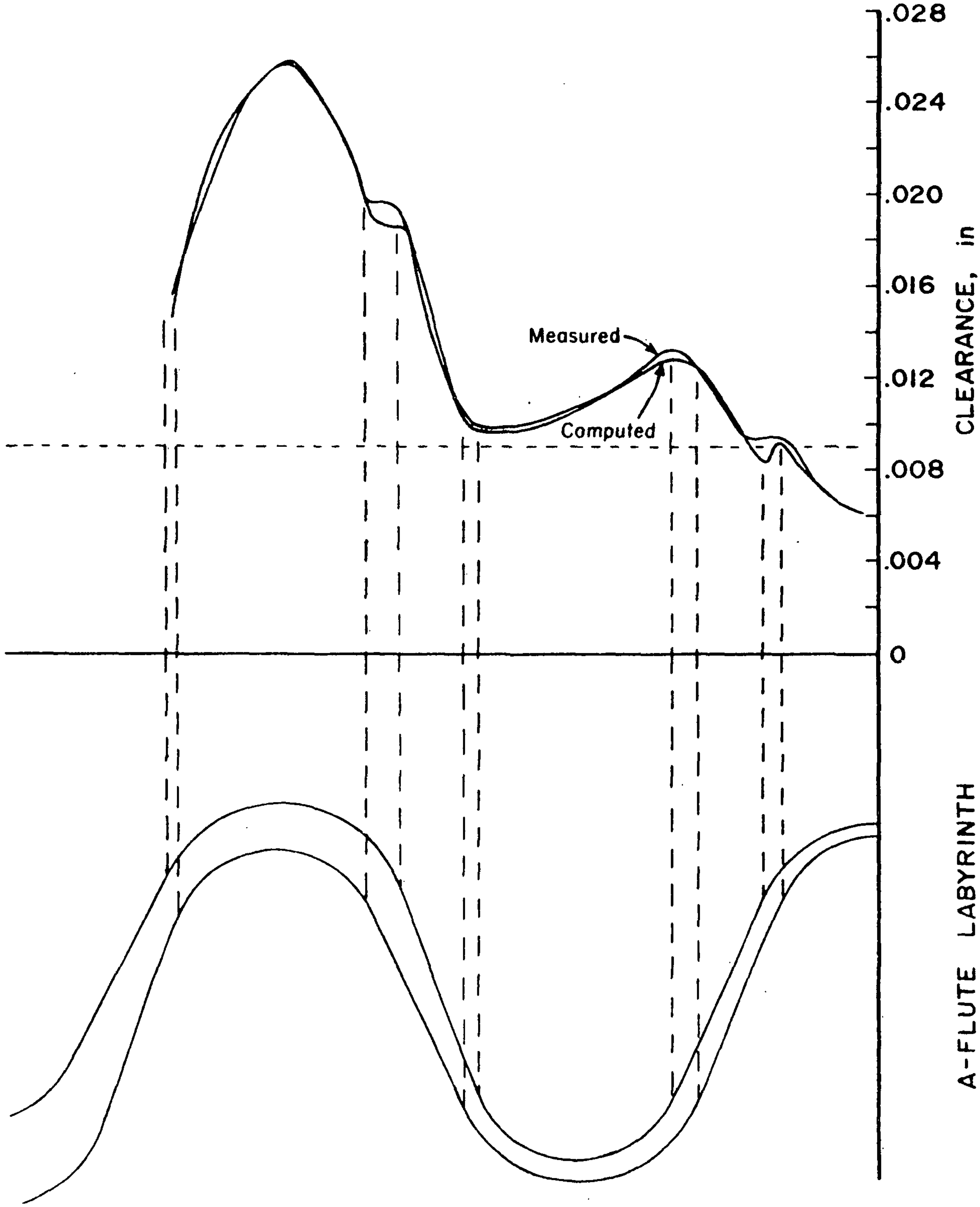

Figure 23. Clearance in Labyrinth.

For this purpose, an Autech Model AKR Laser Dimension gage was used to measure the flute profiles (30). The profiles were measured by passing a web of single-faced board under the laser at a controlled speed. The signal from the laser sensor was analyzed to give the required flute shape information.

Three single faced boards, fabricated using mediums 1, 19, and 114, were analyzed for geometry differences caused by the hot and cold corrugating process. The single faced boards were fabricated at a speed of 200 FPM under normal but controlled conditions of web tension and corrugating pressure. The average flute profiles of the cold and hot forming single faced board in Fig. 24 show that cold and hot formed flutes are very similar in shape. However, both cold and hot formed flutes are unsymmetrical and to about the same degree. The unbonded tip is somewhat flattened and rounds off more gradually to the trailing side than to the drive side. We believe these symmetry differences are related to the different forming conditions for the driven and trailing sides. In addition, the dynamic forces imposed in forming the bonded and unbonded tips are somewhat different as noted in Fig. 7. While the dynamic forces vary in magnitude with corrugator operation, it appears that less molding force is applied to the unbonded tip as a result of the drive action.

We also observed in the flat crush test that the unbonded tip collapses first and more rounding-off occurred on the trailing side. This may be due to the above geometry differences.

Figure 24 shows that the cold formed flutes have slightly higher caliper, as noted in past studies. There were slight indications that, in two cases, cold formed flutes were more symmetrical in shape; in one case, the hot formed flute was more symmetrical.

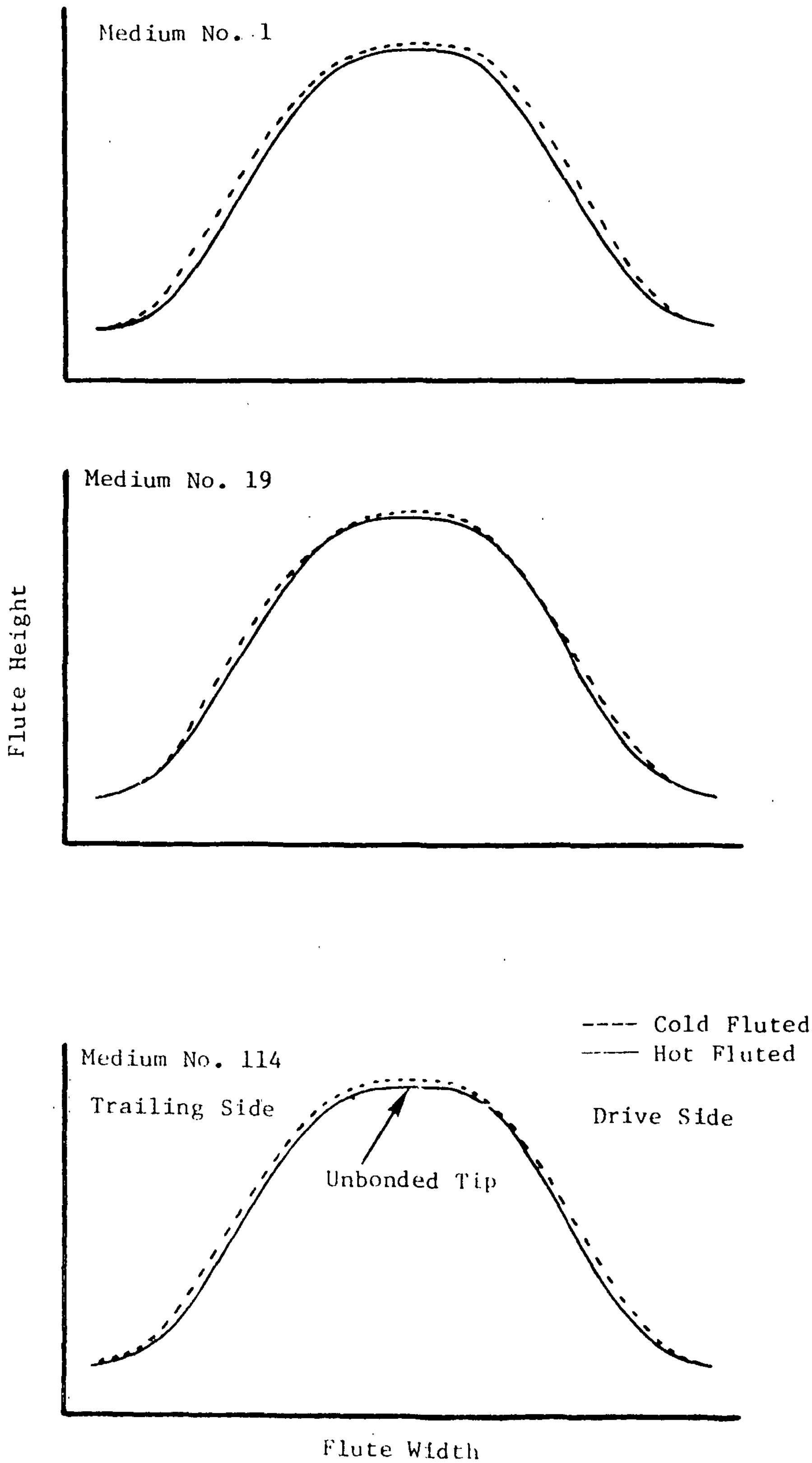

Figure 24. Flute profile shapes.

The effects of flute geometry on board performance (flat crush, flexural stiffness, short column compression, top-to-bottom compression, etc.) have not been theoretically evaluated as yet. Of particular interest is the flat crush performance. Because the hot and cold formed flutes are very similar in geometry, the differences in flat crush performance are more likely due to differences in material properties than to shape.

6. Mechanics of Flat Crush

One of the specific purposes of our forming research has been to determine why some mediums yield ultimate flat crush strengths which are different for hot and cold forming. In the "Background" discussion we noted that forming conditions appeared to have no major effect up to the first peak of the flat crush load-deformation curve (see Fig. 20 for example). Thus hot and cold formed board should respond similarly to normal converting stresses of the flat crush type.

However, some cold formed mediums exhibit lower ultimate flat crush strengths than under hot conditions as shown in Fig. 8. In both cases the mediums deform into a hat-shaped frame (Fig. 13). However, the cold formed medium seems to deform less symmetrically as shown in Fig. 12 and 13. In most cases, the second peak in the load-deformation curve (Fig. 8) is absent or only manifests itself as an inflection in the curve if the cold flat crush is low. We found earlier that such cold formed mediums exhibited lower edgewise compressive strength in the flank/tip regions. It appears that the compressive losses affect the formation of the "plastic" hinge points during crushing and the ultimate load.

Flat crush loads are resisted by the flanks of the flute. Figure 25 shows that flat crush loads (expressed as load per unit length of flute side

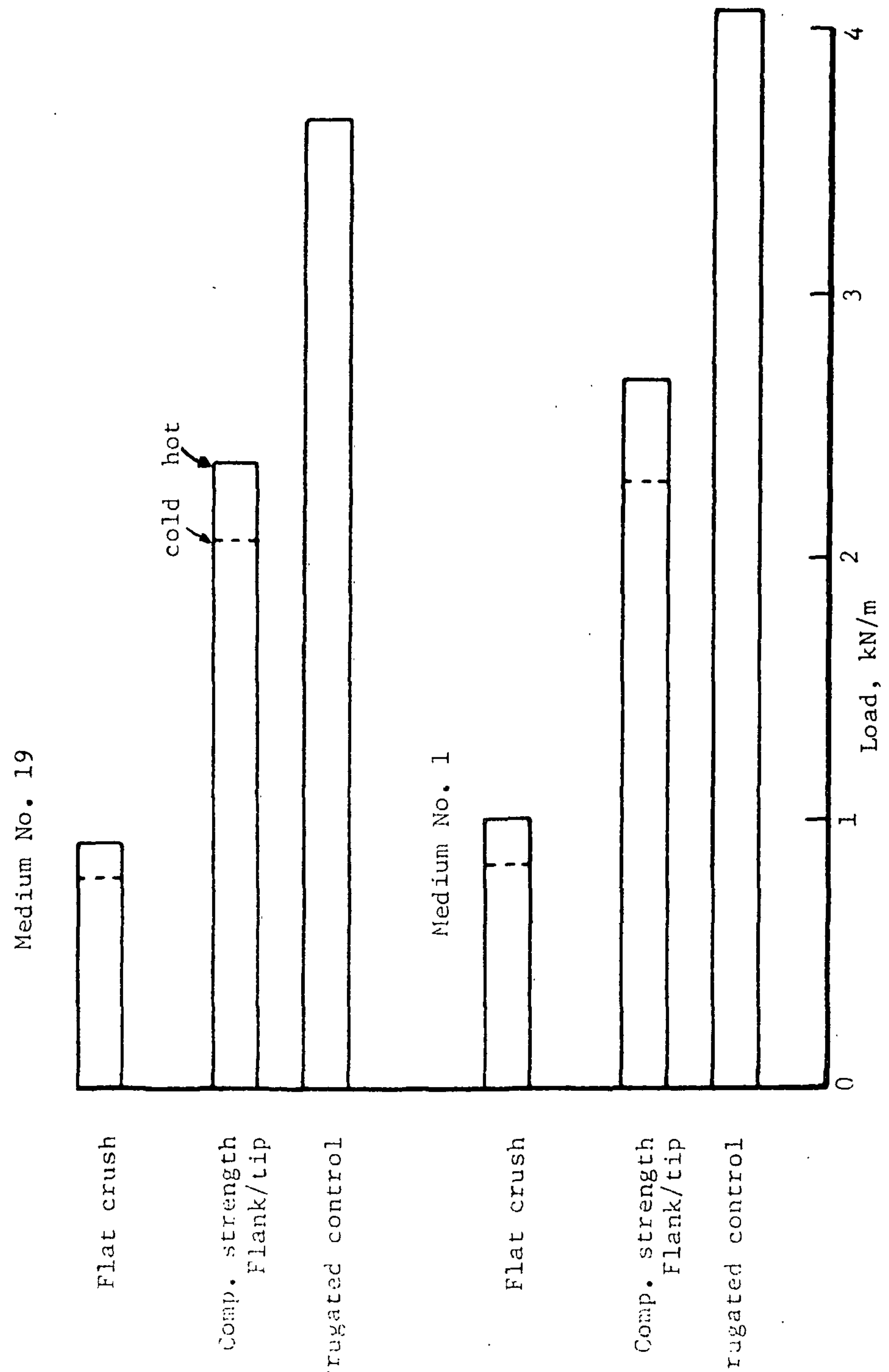

Figure 25. Flat crush and m.d. edgewise compressive strength comparisons.

wall) are substantially lower than the STFI compressive strengths of the uncorrugated medium. They are also lower than the compressive strengths exhibited by the formed mediums in the flank/tip regions. While the flank/tip compressive strengths correlate with the flat crush results (Fig. 11), the differences in magnitude in Fig. 25 indicate that the mechanism controlling flat crush load-deformation behavior needs to be placed on a sound theoretical basis.

Two analytical approaches have been used to develop a better understanding of the flat crush load-deformation curve. The more fundamental approach was directed to developing a finite element model for the flat crush load-deformation curve. The second approach utilized a simple frame analysis as a conceptual way of explaining various aspects of ultimate flat crush behavior.

a. Finite Element Analysis

The initial finite element solution allowed for the large deflection behavior of the sheet structure. However, as a first step the medium properties were considered to be linear-elastic. This model appeared to provide reasonable estimates of the initial slope of the flat crush curve, particularly when transverse shear effects were considered. This model could not adequately describe flat crush loads beyond the first peak load. Among other things, the results indicated that it would be necessary to account for material nonlinearities as well as large deflection. However, this would result in a more complex model. To reduce the modeling costs and time, only five elements over a 1/4 flute length were employed. This results in a crude representation of the flute shape (Fig. 26).

Several different kinds of finite elements were used in the nonlinear structural models: an elastic-perfectly plastic hinge, elastic hinge, elastic beam, elastic-plastic strut, and gap element, as shown in Fig. 12. We had to

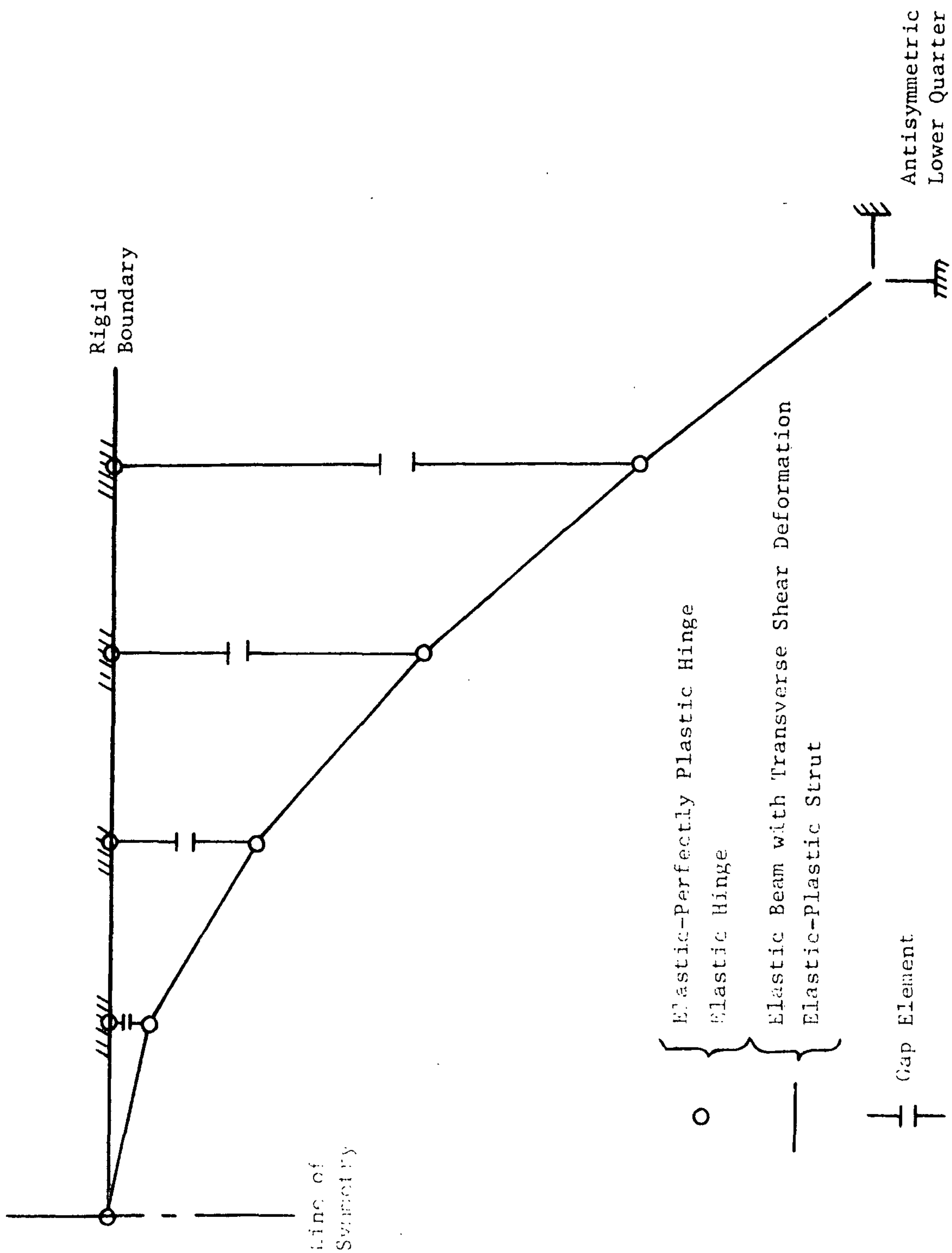

Figure 26. Quarter symmetric/antisymmetric flute model.

utilize several different kinds of finite elements because there was <u>no single finite element available</u> in the element library which incorporated all of the material characteristics needed to model paperboard. The elastic-perfectly plastic hinge and the elastic hinge were used to model the bending characteristics of the medium for large rotations. The elastic beam elements were used to incorporate transverse shear deformation characteristics into the model. The elastic-plastic strut was used to model the in-plane compressive stiffness of the medium. Finally, the gap elements were used to model the effect of the rigid boundary of the liners on the medium shell structure. The characteristics of the elastic-plastic hinge, elastic hinge, elastic beam, and strut were chosen so that compositely they would model the overall material behavior of the medium in a general state of compression. Ideally, it would have been desirable to use a beam element which included all of the necessary material characteristics, i.e., (1) transverse shear; (2) tension/compression; and (3) nonlinear material behavior.

Nonlinear material properties for compression and tension were experimentally obtained (Fig. 27) at standard test conditions. The compressive properties were used to describe the material response of the strut finite elements. Both the tensile and the compressive stress-strain curves (Fig. 27) were needed to compute the elastic-plastic hinge response (Fig. 28). The solid line is the computed nonlinear hinge response. Since the hinge element could only exhibit elastic-perfectly plastic behavior, the response indicated by the dashed line was used in the structural model. The elastic hinge (Fig. 28) was assumed to have a stiffness of 1 inch-lb/RAD. This value needs to be experimentally confirmed for the large rotational regime.

The predicted large deflection response to flat crush loads of the board after including nonlinearities is compared to the experimental results in

Fig. 29. We expected that including the material nonlinearities would significantly lower the load-deflection curve relative to the elastic case. This was confirmed as can be seen by comparing the two dashed curves.

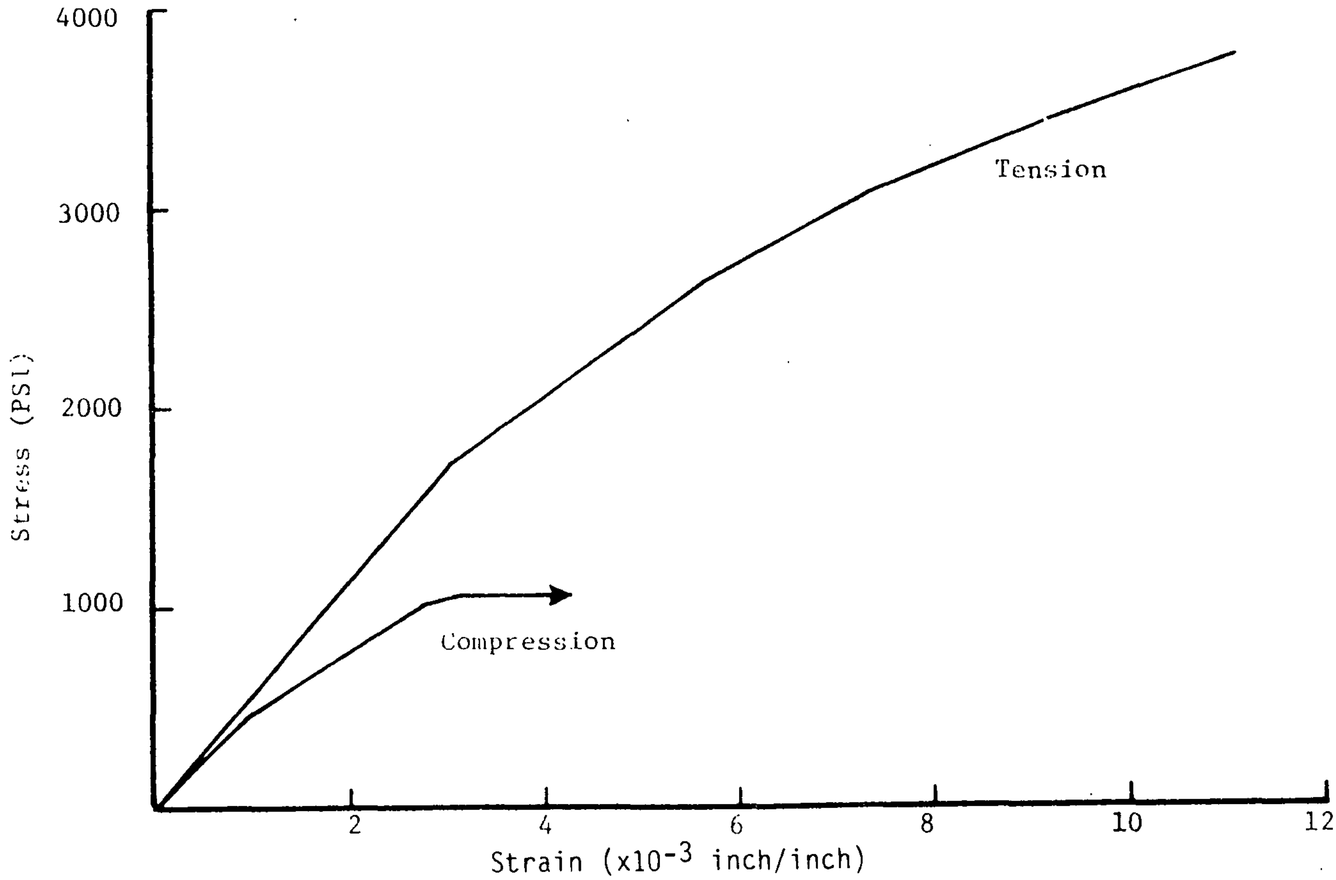

Figure 27. Tensile and compressive stress-strain curves for the uncorrugated medium.

The flat crush response is plotted on a larger scale in Fig. 30. The general shape of the experimental response curve was captured but the local instabilities (i.e., dips) were not. Possible explanations for this include:

1. Difficulties in evaluating all the material properties needed, e.g., the transverse shear load-deformation curve. Also no allowance was made for changes in properties due to forming.

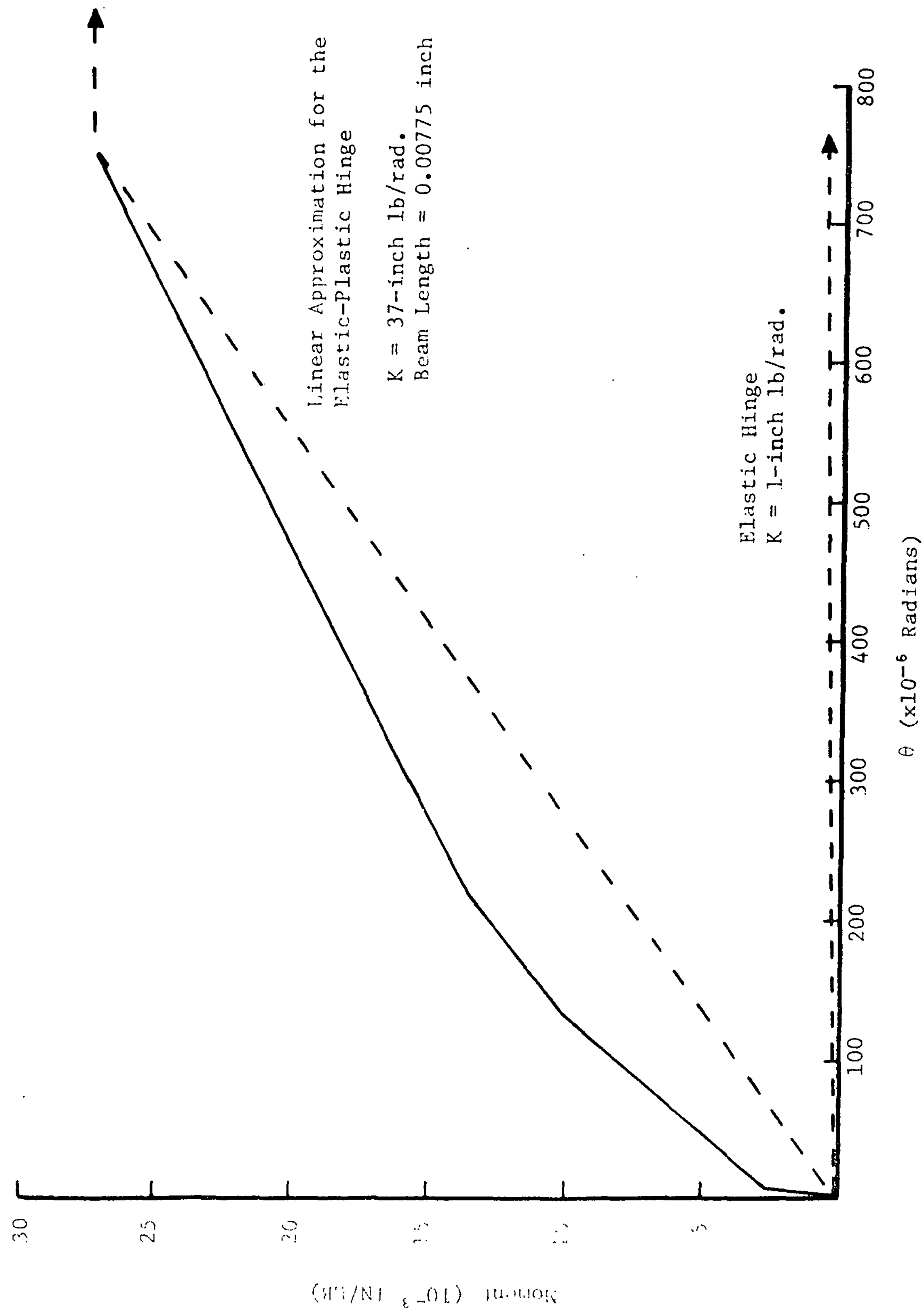

Figure 28. Load-deflection response for the hinges.

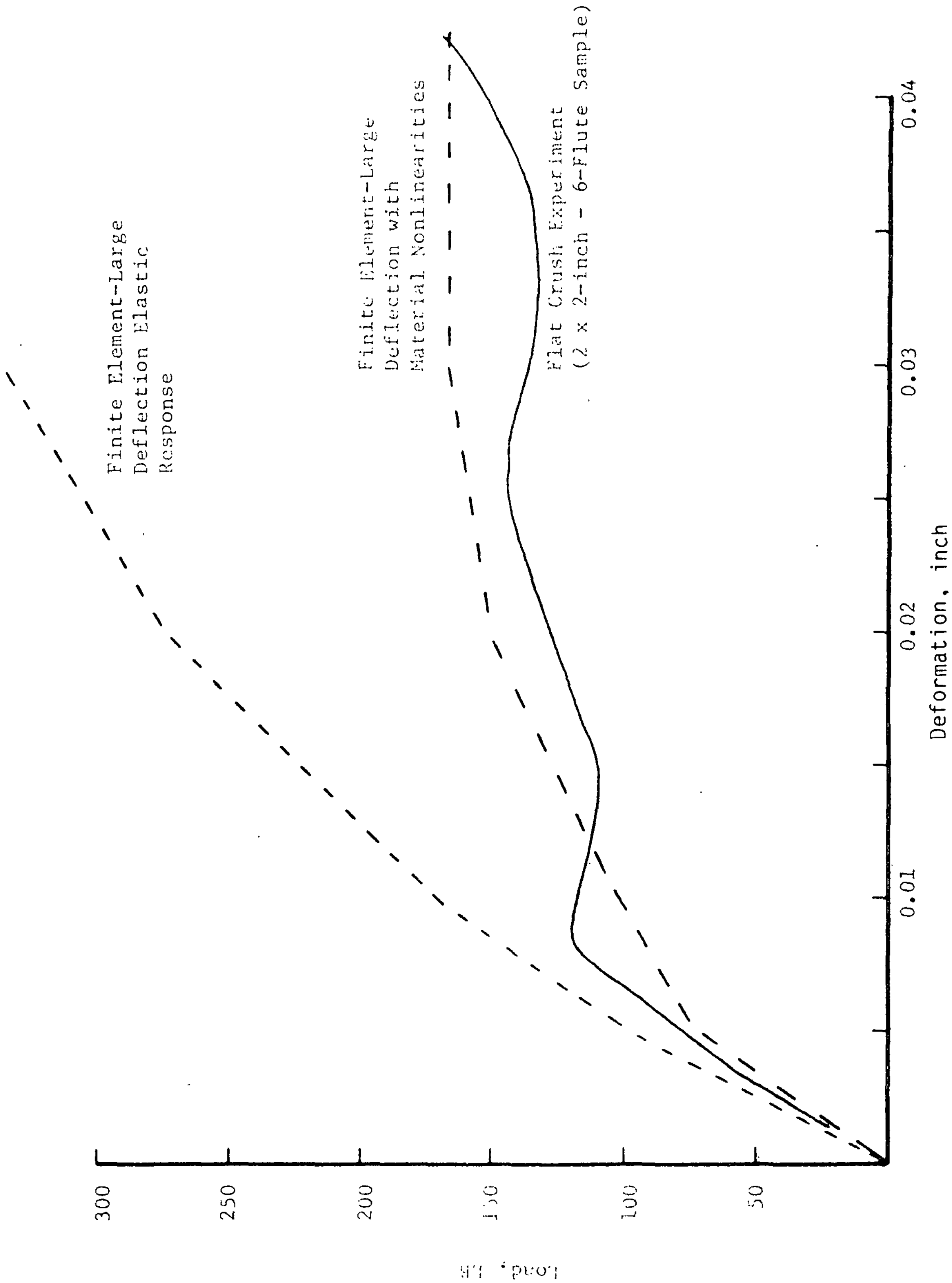

Figure 29. Elastic and nonlinear model load-deflection responses for flat crush.

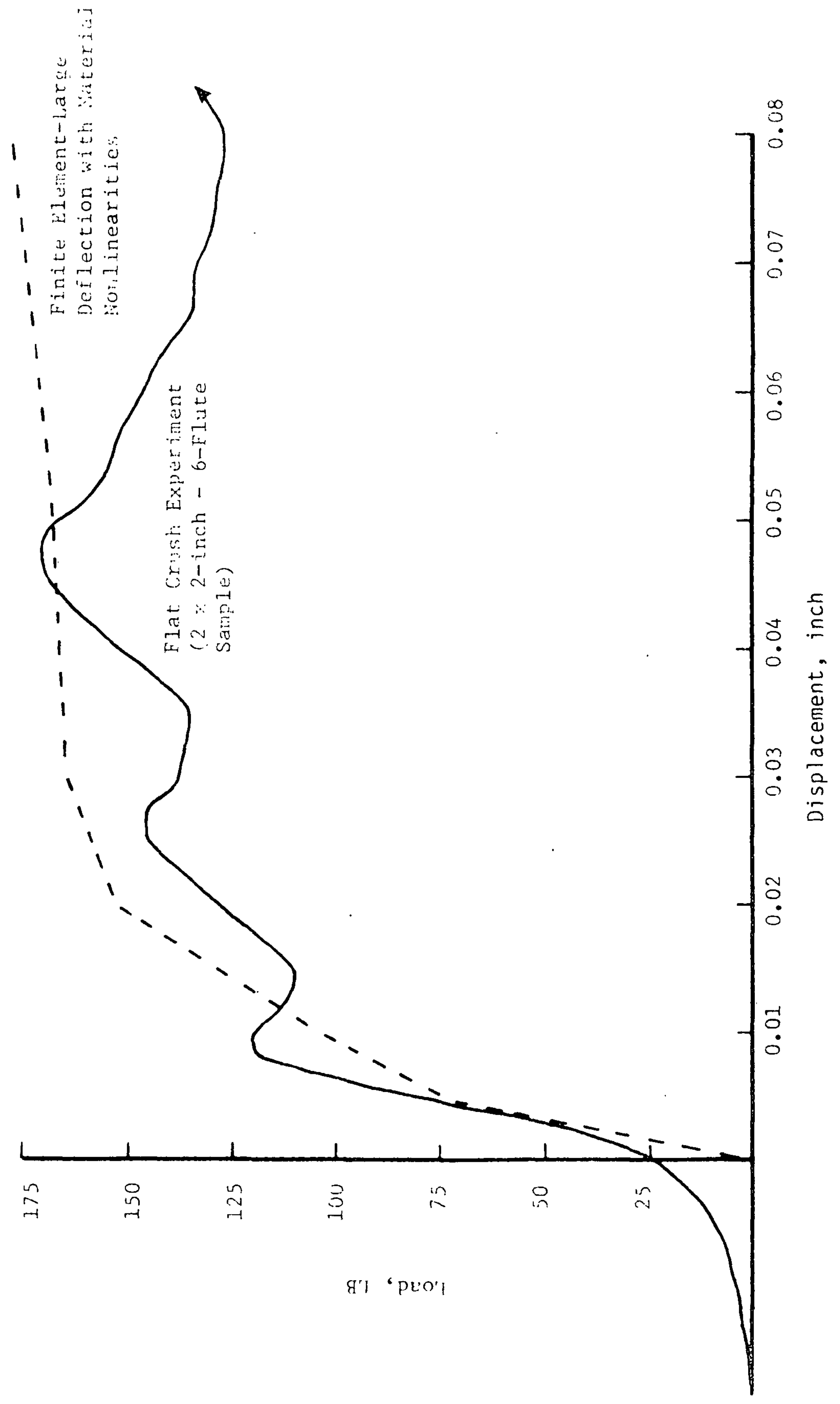

Figure 30. Predicted and measured load-deflection responses for flat crush.

2. Incomplete material description for large rotational behavior.
3. Incomplete understanding of material nonlinearities related to coupling effects between axial stress, bending stress, and transverse shear.
4. Too few finite elements were used: for this first try, the shell structure was divided into only five segments.
5. Stress stiffening as well as large deflection may be needed.
6. The large deflection approach used in this particular computer code is too approximate; another code may be needed.
7. Flute geometry was specified to be a perfect sine wave. A nonsymmetric nonideal flute profile may be needed as described in the flute geometry section.

Another aspect observed in the experiment that was not captured by the finite element model is the almost complete vertical straightening of the flute sides during flat crush. The predicted flute profiles show no pronounced tendency toward this vertical straightening (Fig. 31). However, the simpler linear-elastic, large deflection model captured the vertical straightening (Fig. 32). Note that the deflected shape approximates the observed hat shape of the medium near failure. Currently, we do not know if this difference in behavior is a result of the simplified finite element model (only 5 elements per 1/4 flute), or the material property assumptions.

We believe that Fig. 30 and 32 illustrate the potential ability of finite element models to predict the entire flat crush load-deformation curve. Such models would allow us to better define the characteristics of the medium

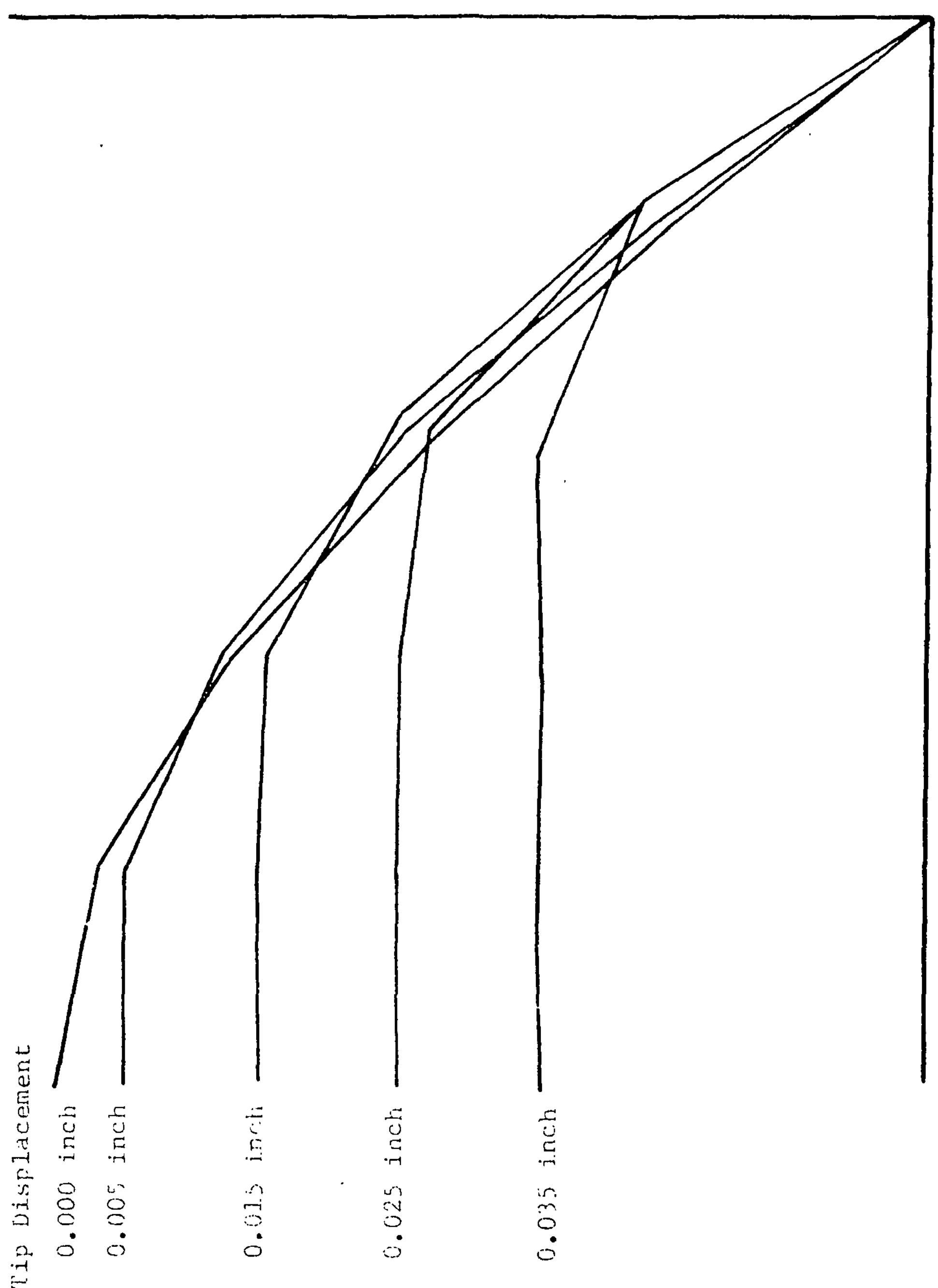

Figure 31. Deflected flute profiles for quarter flute model allowing large deflections and material nonlinearities in analysis.

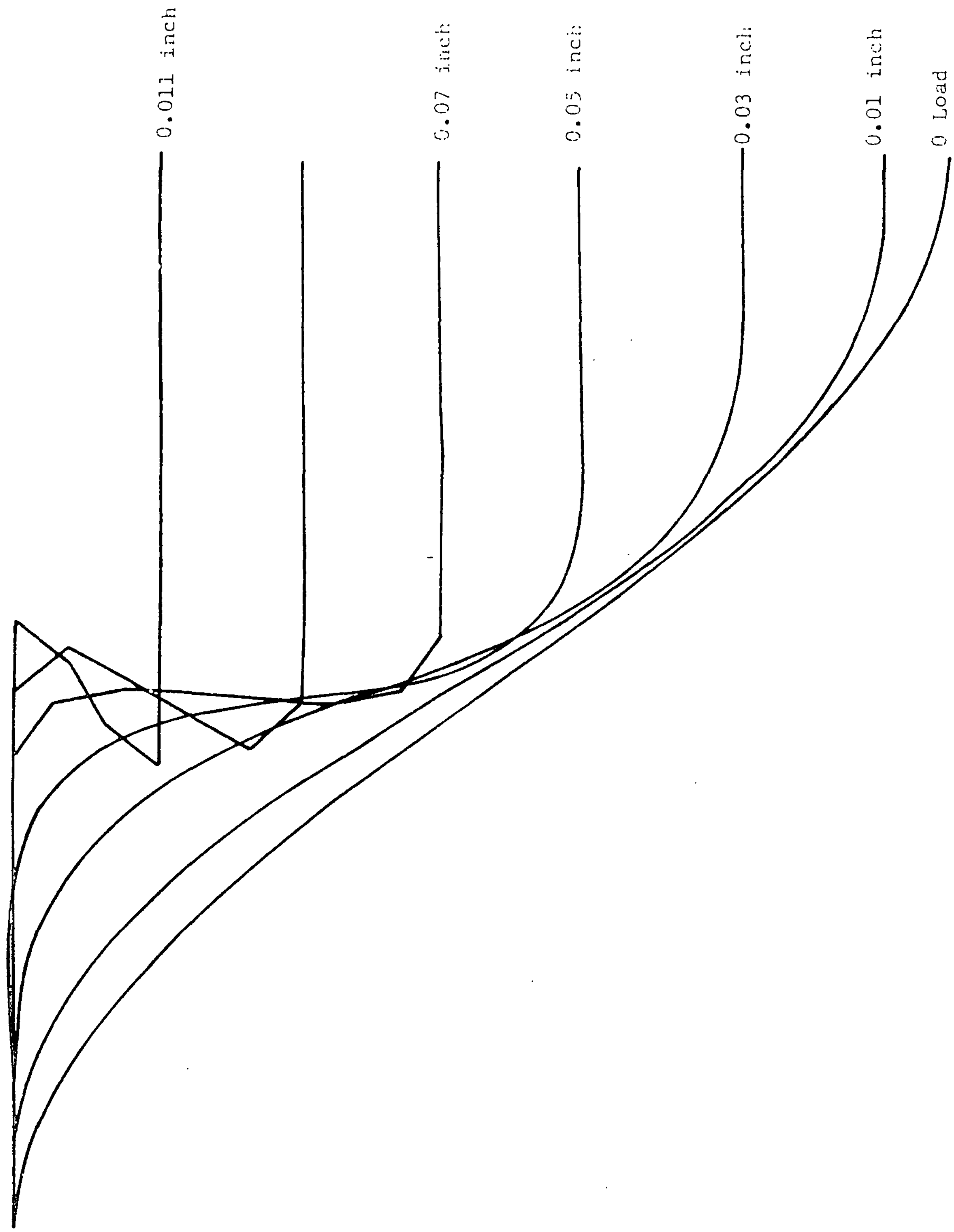

Figure 32. Deflected flute profiles for half-flute (large deflection, linear elastic model).

which govern not only flat crush failure but also the initial load behavior so important in converting. However, the full potentials of finite element analysis elastic, inelastic, and viscoelastic cannot be realized until methods are developed to more completely evaluate the properties of medium and board. For this reason further work on this model has been stopped pending the development of the required material models in ongoining fundamental studies at the Institute.

b. Frame Analysis

In the final stages of the flat crush test the medium forms a frame-shaped structure (Fig. 33). Timoshenko (31) discusses a frame buckling case which is somewhat similar to flat crush. This results in an Euler type column equation where the end-condition coefficient depends on the thickness of the medium and the frame dimensions. Thus, $P = k^2 E I/\ell^2 = k^2 Et (t/\ell)^2$

where P = maximum load,

E = modulus of elasticity in direction of load,

I = moment of inertia, = $wt^3/12$,

w = column width,

t = thickness,

ℓ = frame height, and

k^2 = frame coefficient dependent on the lengths of the elements and moments of inertia (I, and I_1, in Fig. 18).

The frame equation provides a conceptual way to explain such effects as flute geometry, the weight or thickness of the medium and various papermaking factors. Brecht and Bachmayer (32), in an extensive investigation, showed that "generally everything which increases the elastic modulus" increases Concora strength. Their work was carried out on hot formed medium but we believe cold formed mediums will respond in a similar way to many of the papermaking conditions studied. They also noted that hot Concora strength was quite sensitive to thickness. They cited the following: (1) Concora may not be greatly affected by wet pressing if the increases in modulus are counterbalanced by the decreases in thickness; (2) dry pressing can reduce Concora because the reductions in thickness are not compensated by real increases in fiber-to-fiber bonding; and, (3) increasing the fiber orientation increases the elastic modulus and Concora in the m.d. direction. Brecht and Bachmayer proposed an empirical modification of Eq. (1) which related Concora to the modulus, basis weight, and the square of the thickness. The relationship explained the general trends but there was considerable scatter, indicating other factors are involved.

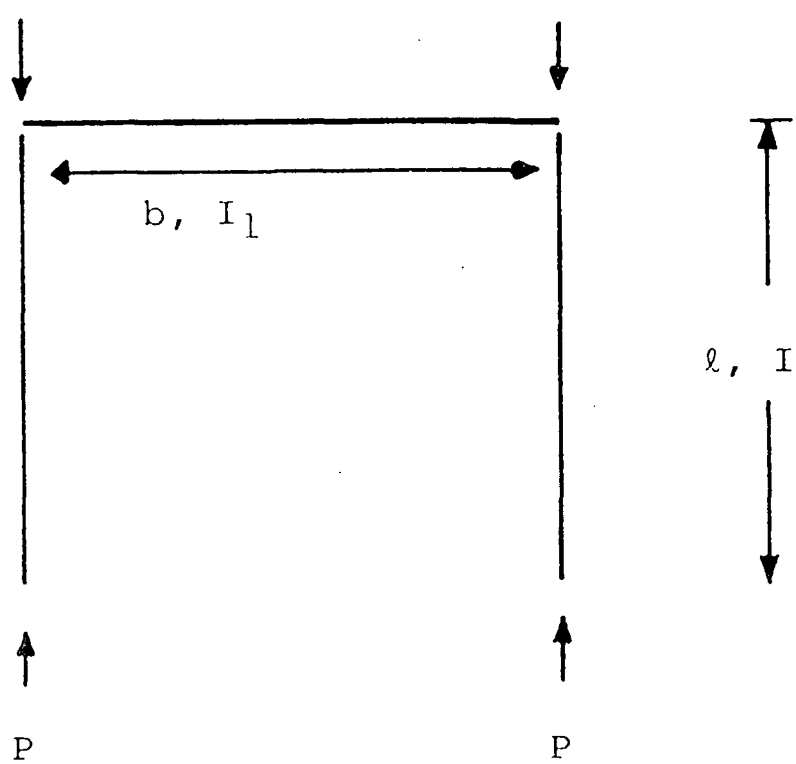

Figure 33. Frame buckling - lateral displacements permitted.

There are theoretical and measurement difficulties in directly applying the frame analysis approach. Among the measurement factors are the determination of (1) effective caliper and (2) modulus after forming. Theoretical difficulties involve allowance for inelastic and/or shear affects, proper evaluation of end conditions, etc.

With these reservations, Fig. 34 shows preliminary frame equation estimates for medium sample 1 in comparison to flat crush and compressive strength. The frame loads are close to the flat crush magnitudes at reasonable frame heights - i.e., 0.060 to 0.075 inch (about 1.5-2.0 mm). They do not necessarily explain cold/hot flat crush differences because it is not possible at present to obtain Et values after forming.

The buckling coefficients in Fig. 34 were based on dimensions scaled from test photographs. There is an indication that hot-formed board has higher k^2 values than cold formed board. This would promote higher flat crush for the hot board.

It is believed the frame equations provide a conceptual way to explain the effects of variables such as flute geometry, medium thickness or weight, etc., on flat crush. However, to explain differences in flat crush due to forming conditions would require adjustment of either the geometrical or material properties in whatever theoretical approach is pursued.

In this connection 34 commercial mediums were fabricated into single-faced board on the Institute's experimental corrugator. Both cold and hot corrugating conditions were employed and the single-faced boards obtained at 600 fpm were evaluated for flat crush. The uncorrugated mediums were evaluated for a wide array of physical properties. The correlations between the uncorrugated medium properties and flat crush are summarized in Table XV.

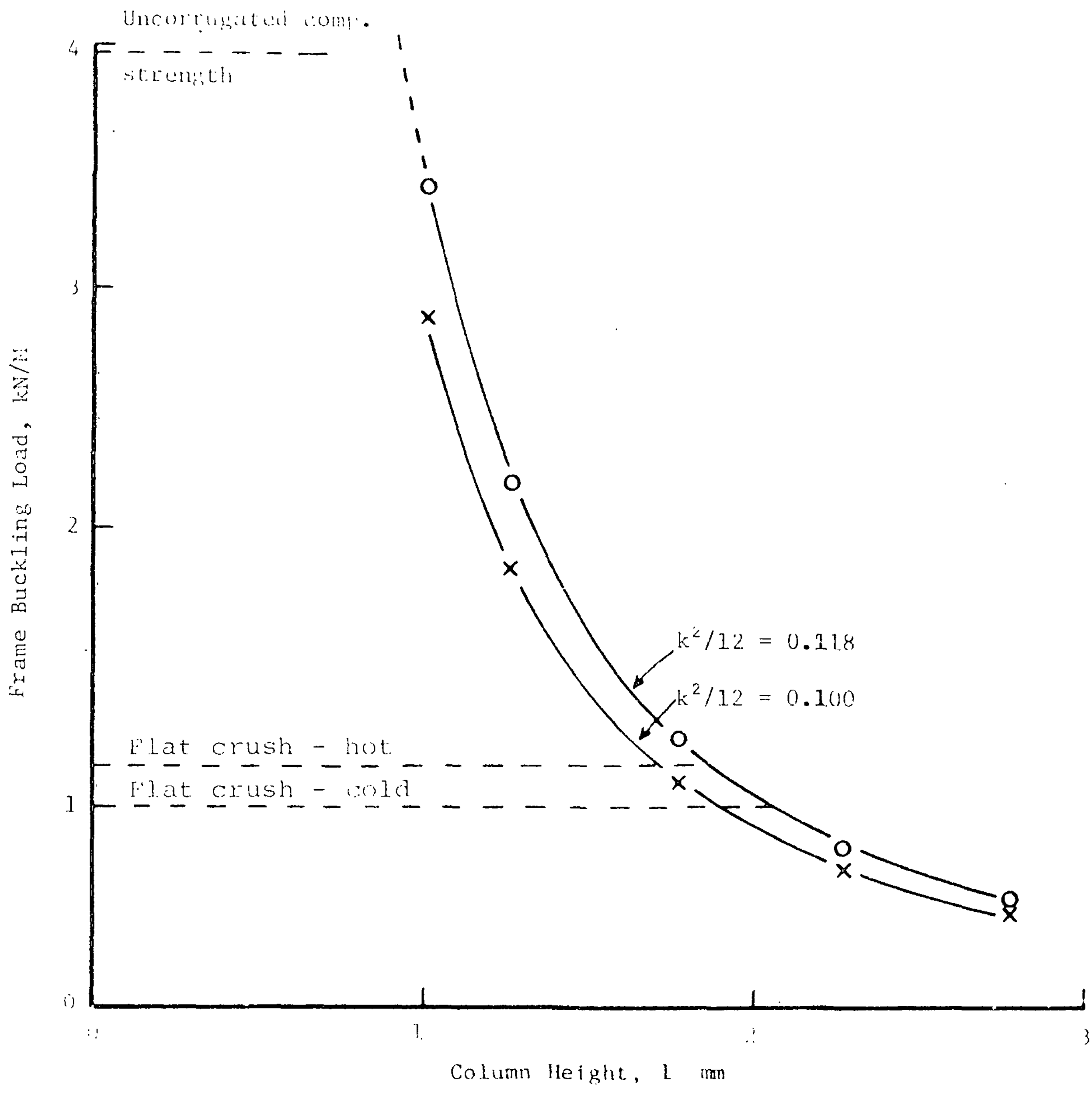

Figure 34. Frame results for various column heights at constant length to width ratios.

TABLE XV

FLAT CRUSH VS. VARIOUS MEDIUM PROPERTIES - 34 MEDIUM SAMPLES

Property	Correlation Coefficient: Hot Flat Crush	Correlation Coefficient: Cold Flat Crush (WTA)
Basis weight	0.21	0.34[a]
Caliper	0.07	0.12
Density	0.08	0.09
Moisture content, cond.	0.19	0.25
Moisture content, uncond.	0.03	0.19
Porosity	-0.25	-0.21
Smoothness	0.08	0.06
Friction, F.S. (cold)	0.11	0.46[b]
Friction, W.S. (cold)	0.18	0.49[b]
Concora	0.94[b]	0.60[b]
Mullen	0.42[a]	0.28
CD Ring	0.59[b]	0.51[b]
MD tensile	0.60[b]	0.47[b]
MD stretch	0.32	0.16
MD TEA	0.46[b]	0.37[a]
MD strecth	0.42[a]	0.41[a]

[a]Significant at 0.05 level.
[b]Significant at 0.01 level.

Under hot corrugating conditions the Concora (hot forming) results were highly correlated to flat crush as would be expected. However, the Concora correlation declined from 0.94 for hot corrugating to 0.60 for cold corrugating. Thus the Concora test, which is based on a hot fluting action, is well correlated to hot corrugating but not to cold corrugating.

Some of the other properties were correlated to flat crush but the correlations were not very strong.

7. Summary

Our research has focused on several areas. They include the effects of hot and cold conditions on (1) fluted medium characteristics, (2) flute geometry, (3) flat crush load-deflection behavior and (4) the effect of various forming stresses on the compressive strength of the fluted medium and (5) the effect of nonlinear properties on combined board strength.

Our results indicate that edgewise compressive strength and other properties of the medium are greatly reduced by both forming processes. The reductions in medium strength are substantial and affect the quality of combined board to about the same extent under either cold or hot corrugating conditions. The reductions in strength are caused by the high bending and tension stresses induced in the medium during forming. For some mediums the strength reductions are somewhat more severe under cold corrugating conditions, and this reduces the ultimate flat crush strength. However, the initial flat crush load-deflection behavior is the same for both cold and hot forming, only the failure loads are different; therefore, cold and hot formed boards should respond in the same way to normal converting. Furthermore, the strength reductions caused by both forming processes are much more important as a subject for study than are the differences in strength between hot and cold formed structures. The work done concerning medium properties indicates substantial improvement can be obtained with regard to these strength reductions.

Specific findings are noted below.

(1) The edgewise compressive strength of the fluted medium (cold or hot formed) is reduced in both m.d. and c.d. directions. The reductions range from 35-50% in the m.d. direction. The c.d. compressive strengths are reduced by about 20-30%. These findings are significant

because they indicate that the fluting process degrades the compressive strength potentials of the medium in both directions. Thus box compressive strength, flat crush strength, and other combined board properties are all reduced.

(2) Some cold formed mediums show more evidence of compressive strength reduction in the flank/tip regions than under hot conditions. We believe this accounts for the lower flat crush obtained with some cold formed mediums. It was also noted that such cold formed mediums tend to exhibit more compression degradation on the trailing flank than on the driven flank.

(3) The transverse bonding strength of the medium is also reduced by forming in both the cold and hot processes. The reductions in bonding would be expected to reduce the compressive strength and other properties of the formed medium.

(4) In simulation experiments, we observed that prestressing the medium in bending reduces edgewise compressive strength. Our results also show that the compressive losses are increased by higher tensions during bending. As expected, greater losses occur as the radius of bending decreases because the strain in the outer fiber layers is inversely related to the radius. These results suggest that the compressive strength losses - and hence the

flat crush losses - are due primarily to bending stresses induced during forming.

(5) Analysis of clearances in the labyrinth shows that a potential pinch point exists about 1/2 flute ahead of the center line. In past work at the Institute, high speed motion photography shows that fracturing occurs before the medium reaches the center line - also by about 1/2 flute. If the medium cannot freely slide past the "pinch point" it will be strained more highly and this will increase the risk of fracture or cause greater reductions in strength of the medium. Drives for both corrugating rolls (dual drives) have been tried at the Institute and elsewhere in the past to minimize clearance problems and improve fluting. However, in limited trials dual drives did not produce marked improvements in fluting performance.

(6) When the medium is formed around the flute tip the severe bending stresses are relieved by the simultaneous shear strains. Exploratory evaluations indicate the m.d. transverse shear modulus of medium is 30-40 times lower than the m.d. extensional modulus. Because of the low shear modulus, shear effects can be an important factor in allowing the medium to conform to the fluted contour. Because the shear characteristics of the medium are important in corrugating, one aspect of future work should be the development of methods for evaluating and

controlling this property. We would then be able to account for both bending and shear effects in corrugating and other forming operations in board conversion.

(7) A thorough review of flat crush technology was carried out. It appears that the initial portion of the flat crush load-deformation curve is critical in determining whether crushing in finishing will degrade board quality. However, the entire load curve is important because field performance depends on crush resistance up to ultimate failure.

(8) Detailed analyses of hot and cold formed board indicate there are only small differences in profile shape. Thus, differences in the ultimate flat crush performance between hot and cold formed board are due to fluted medium characteristics rather than flute geometry.

(9) We also carried out a preliminary mechanics analysis of flat crush load-deflection behavior using finite element techniques. The finite element models appear to predict the general load response of the flute. The models also show promise of predicting the deflected shape as loading progresses. Among other things the initial results suggested that the shear characteristics of the medium may be of importance. However, the full power of finite element modeling cannot be brought to bear until we have completed development of methods of evaluating medium properties.

D. HIGHER PERFORMANCE EXPERIMENTAL CORRUGATING MEDIUMS

1. Background

Past work has established that medium properties affect and are affected by fluting. It has also been shown that flat crush and ECT potentials of corrugated board, which are dependent on the medium, are greatly reduced by the fluting process. These reductions in strength are caused by the high bending and tension stresses induced in the medium during fluting. Both the in-plane Young's modulus and the transverse shear modulus of the medium affect the losses. With this background a study was instituted to optimize medium properties to prevent strength losses in fluting and, hence, improve performance.

In either the cold or hot fluting process, about 40% of the MD and 20% of the CD compressive strength of the medium are lost. Reducing these losses would yield significant savings in the cost and energy associated with the manufacture of medium.

The losses in compressive strength are dependent on three factors: (1) the basic properties of the medium; (2) the temporary alteration of these properties by preconditioning or pretreatment on the corrugator; and, (3) the design and operation of the corrugator. Retention and improvement of medium strength can be obtained by changing the base properties of the medium or by improvements in preconditioning or pretreatment. Work in the latter area is underway for the FKBG.

The work on improving medium properties is following these steps:

1. selection of medium property targets,
2. selection of papermaking approaches,

3. laboratory and pilot corrugator trials, and
4. economic assessment.

The basic goal is to increase the compressive strength of the corrugated (fluted) medium in both directions, while improving runnability. Such improvements will increase flat crush and the contribution of the medium to ECT without losses in corrugator productivity.

Compressive strength is highly related to the elastic moduli of the sheet. High compressive strength is favored by high moduli in the plane of the sheet (E_x and E_y) and by high transverse (out-of-plane) moduli (E_z, G_{xz}, and G_{yz}) (29). Densification to increase fiber bonding is an effective way to increase all of these moduli and, hence, compressive strength. The forming behavior of medium is mainly dependent on the MD properties of the sheet and friction. As we increase compressive strength, however, we must make sure that the formability of the medium is not harmed.

Past work indicates that perhaps 1/3 to 1/2 of the forming losses can be avoided. Results on densified, experimental oriented sheets, made on the Formette former, show substantially higher flat crush strengths and lower compressive strength losses during fluting than a commercial 26-lb medium, thus confirming that large improvements are possible.

2. Experimental Mediums with Improved Properties

Oriented sheets were made on the Formette and the pressing and drying conditions were varied to change the density. For these trials, a semichemical stock was obtained and defibered and refined at the Institute to about 350-400 CSF. For sheet making purposes, this was blended with 25% of a softwood kraft refined to about the same level. The sheets were made to a nominal weight of 26 lb/1000 ft (127 g/m^2). Corrugating trials were made by splicing the experimental sheets into a carrier medium and making single-faced board.

For the initial trials, the Formette sheets were pressed and dried on a belted drum press dryer. Both cold and hot pressing conditions were used, in each case followed by a ten minute hot drying period under the belt against the dryer. At each pressing/temperature condition the number of passes through the press nip was varied to give three levels of densification. Thus, the initial trial (Trial I) involved preparation of sheets representing six different pressing/drying conditions. A completely independent set of sheets was prepared, duplicating the Trial I conditions insofar as possible. These are termed Trial II experimental sheets in the following discussion.

Figure 35 shows that the experimental sheets from both trials exhibited both higher retention ratios and compressive strengths than 26-lb commercial mediums fabricated into single-face board and evaluated in the same way. The commercial mediums were fabricated into single-faced board at the same time and under the same conditions as the experimental mediums. Retention ratio is the ratio of the STFI compressive strength on the flanks of the formed flutes to the compressive strength of the unformed medium. Thus, the experimental medium base strength exhibited significantly less loss in compressive strength due to fluting than the commercial mediums. The higher retention ratios suggest that the same strengths can be achieved with less fiber. Note, also, that the experimental mediums representing the independent Trials I and II behaved about the same, despite the many variables involved in preparation, forming, pressing, and testing.

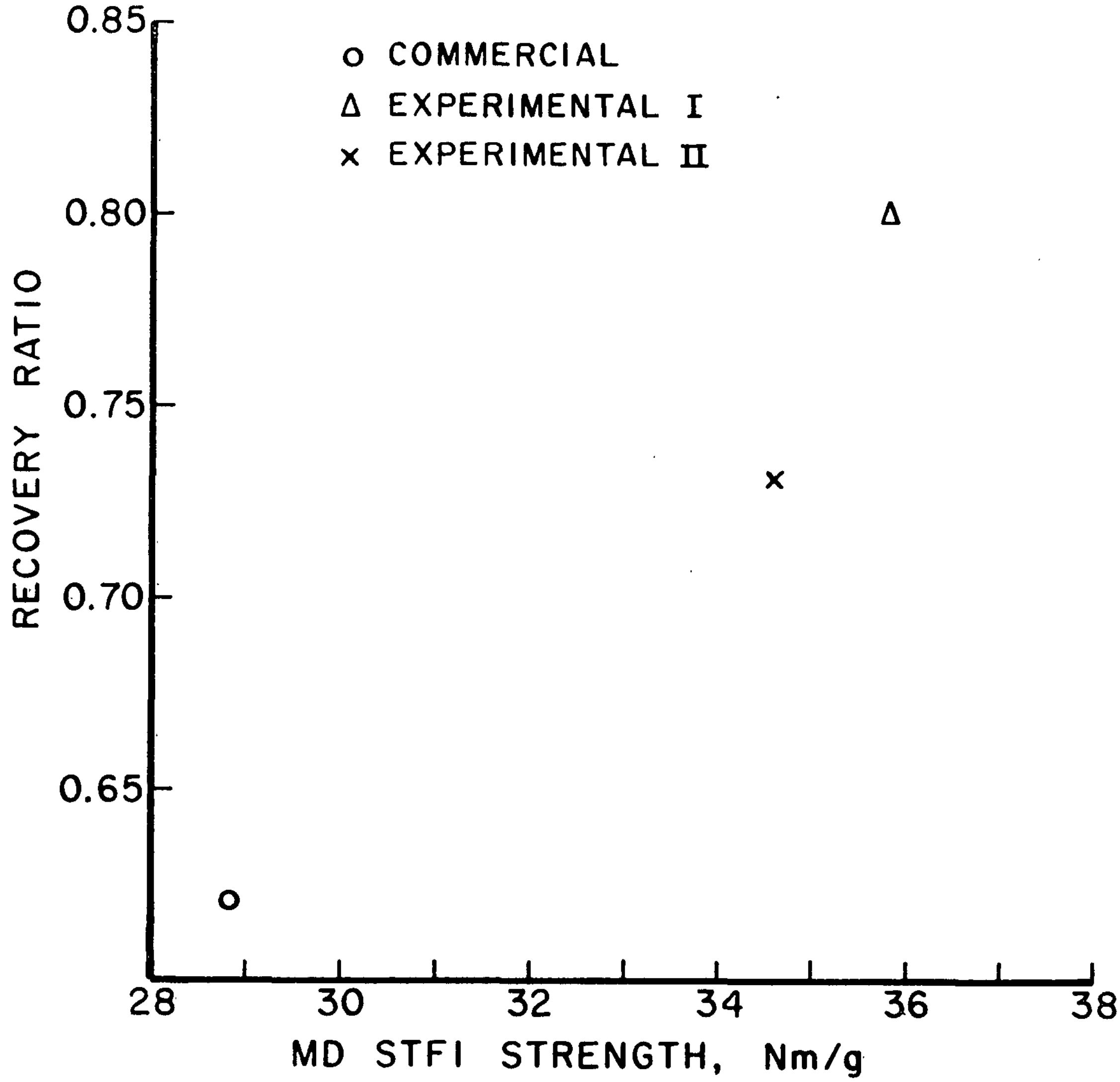

Figure 35. Compressive strength retention ratios of experimental and commercial mediums.

Figure 36 shows that the flat crush strengths of the experimental mediums were about 37% higher than the flat crush strengths of the commercial mediums at a Concora level of 60 lb (fairly typical of current commercial medium). The higher flat crush strengths of the boards prepared from the experimental mediums are due to the combined effects of higher compressive strength in the unformed state and higher retention ratios in the fluting operation.

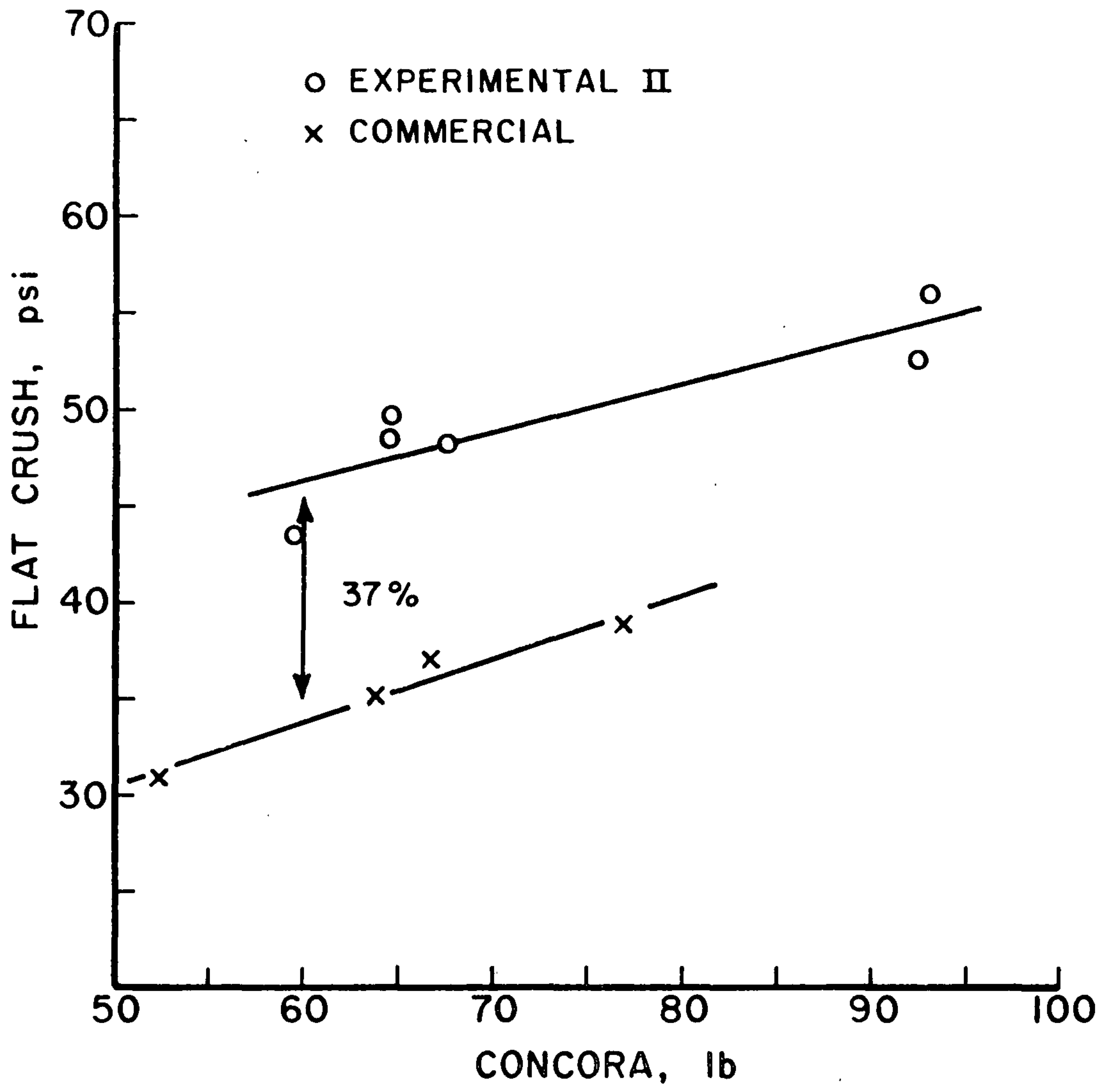

Figure 36. Flat crush vs. Concora results for experimental and commercial mediums.

Figure 36 also shows that the Concora test failed to predict the higher flat crush performance of the experimental mediums. To check on this, the results for the four mediums used as controls in Fig. 36 were compared with previously obtained results on 35 commercial mediums (Section E). As seen in Fig. 37, the flat crush vs. Concora results for the controls follow the same relationship as

TABLE XVIII

PROPERTIES OF SINGLE FACE BOARD FROM COLD AND HOT PROCESSES

Code	Caliper, mils Cold	Caliper, mils Hot	Draw Factor Cold	Draw Factor Hot	Flat Crush, psi Cold	Flat Crush, psi Hot	Pin Adhesion, lb/8 inch^2 Cold	Pin Adhesion, lb/8 inch^2 Hot	Edgewise Compression CD, lb/inch Cold	Edgewise Compression CD, lb/inch Hot
1	156.3	154.1	1.448	1.445	27.8	34.6	92	77	28.2	28.4
2	157.0	154.4	1.444	1.446	28.2	35.7	99	76	30.0	28.6
3	156.6	154.4	1.440	1.448	28.7	34.1	103	74	31.5	29.5
4	156.9	154.1	1.448	1.446	31.6	34.7	134	91	32.5	29.2
5	154.9	153.6	1.441	1.441	24.5	30.9	99	70	28.1	27.7
6	155.6	153.3	1.448	1.447	28.5	34.1	114	79	30.3	29.6
7	155.4	153.9	1.452	1.448	20.3	21.8	111	65	27.0	25.8
8	156.0	153.4	1.448	1.436	27.9	31.3	111	65	28.6	29.6
9	156.8	154.3	1.449	1.440	27.3	34.5	103	57	28.9	27.9
10	156.6	154.5	1.439	1.438	28.6	33.8	107	61	29.8	28.5
11	157.4	154.1	1.450	1.443	36.5	38.1	129	74	29.1	29.6
12	156.7	154.4	1.442	1.447	24.4	30.7	119	69	26.7	27.2
13	155.8	155.6	1.433	1.443	26.4	30.2	85	50	27.6	28.8
14	157.5	155.8	1.448	1.449	29.4	34.9	101	64	27.2	28.1
15	156.4	155.1	1.437	1.439	28.5	29.5	112	81	29.6	30.0
16	157.2	155.8	1.450	1.448	27.4	31.4	100	92	28.5	28.8
17	156.2	153.6	1.444	1.436	31.2	38.0	135	104	31.6	30.0
18	155.8	153.9	1.435	1.446	28.9	35.7	100	88	32.3	29.7
19	154.9	151.8	1.430	1.436	27.0	28.5	102	83	28.4	28.4
20	--	152.2	--	1.430	--	29.2	--	88	--	28.4
21	156.1	154.6	1.434	1.430	27.2	39.4	96	68	29.4	30.0
22	155.0	152.1	1.433	1.431	28.6	33.9	113	83	30.6	31.2
23	--	152.7	--	1.423	--	35.3	--	97	--	30.4
24	157.1	154.7	1.440	1.438	27.7	29.1	108	63	28.5	28.9
25	155.4	153.6	1.439	1.430	33.9	35.9	121	60	29.5	30.7
26	155.6	154.3	1.432	1.434	30.9	33.5	94	69	27.0	28.5
27	156.9	153.1	1.450	1.427	35.9	39.0	120	73	31.5	30.9
28	157.0	154.8	1.435	1.430	27.8	31.7	105	66	29.2	30.1
29	156.5	154.6	1.442	1.438	31.4	35.3	106	73	30.2	29.4
30	156.8	155.3	1.440	1.442	33.5	34.0	133	77	29.0	29.2
31	155.1	153.7	1.441	1.441	26.8	32.6	117	66	28.5	29.1
32	157.2	154.9	1.443	1.441	29.7	30.7	106	56	29.6	29.7
33	157.0	154.6	1.439	1.436	27.9	29.4	121	92	29.8	30.4
34	156.9	156.2	1.432	1.442	30.9	28.8	87	51	28.3	30.7
35	155.6	155.0	1.433	1.438	28.3	28.8	96	73	29.2	31.2
Mean	156.31	154.18	1.441	1.439	28.89	32.83	108.45	73.57	29.28	29.26
Coeff. of Variation, %	0.5	0.7	0.4	0.5	11	11	12	18	5.0	4

All tests are 600 fpm.
All cold tests with MFA.

TABLE XIX

EFFECTS OF MEDIUM TREATMENT ON PROPERTIES

Code	Caliper, mils		Draw Factor		Flat Crush, psi		Pin Adhesion, lb/8 inch^2		Edgewise Compression CD, lb/inch	
	MTA	w/o MTA	MTA	w/o MTA	MTA	w/o MTA	MTA	w/o MTA	MTA	w/o MTA
1	156.3	156.7	1.448	1.446	27.8	26.3	92	97	28.2	28.0
2	157.0	156.6	1.444	1.436	28.2	26.3	99	98	30.0	30.7
3	156.6	156.8	1.440	1.437	28.7	27.4	103	107	31.5	30.4
4	156.9	156.1	1.448	1.438	31.6	31.5	134	127	32.5	32.1
5	154.9	155.4	1.441	1.425	24.5	25.5	99	96	28.1	27.9
6	155.6	155.9	1.448	1.449	28.5	27.2	114	111	30.3	30.9
7	155.4	155.4	1.452	1.449	20.3	19.2	111	117	27.0	25.0
8	156.0	155.6	1.448	1.447	27.9	28.5	109	112	28.6	29.3
9	156.8	155.4	1.449	1.447	27.3	29.7	103	111	28.9	28.5
10	156.6	155.8	1.439	1.435	28.6	29.3	107	111	29.8	29.5
11	157.4	157.0	1.450	1.447	36.5	33.5	129	127	29.1	30.9
12	156.7	155.9	1.442	1.450	24.4	25.4	119	112	26.7	27.3
13	155.8	156.0	1.433	1.434	26.4	25.2	85	83	27.6	27.3
14	157.5	156.7	1.448	1.443	29.4	30.3	101	107	27.2	27.6
15	156.4	154.2	1.437	1.416	28.5	29.1	112	102	29.6	27.5
16	157.2	156.9	1.450	1.449	27.4	27.7	100	107	28.5	26.7
Mean	156.4	156.0	1.4452	1.4405	27.88	27.63	107.3	107.8	28.98	28.72
Coeff. of Variation, %	0.5	0.5	0.4	0.7	12	12	12	10	5.5	6.6

All tests are 600 fpm.

previously obtained for commercial medium. In contrast, for a given Concora, the experimental mediums exhibit much higher flat crush values than the commercial mediums. The Concora test clearly does not simulate all aspects of a commercial corrugating operation, e.g., preconditioning, tension, pressure, etc. Apparently, these differences contribute to the inability of the Concora test to predict the performance of the experimental mediums.

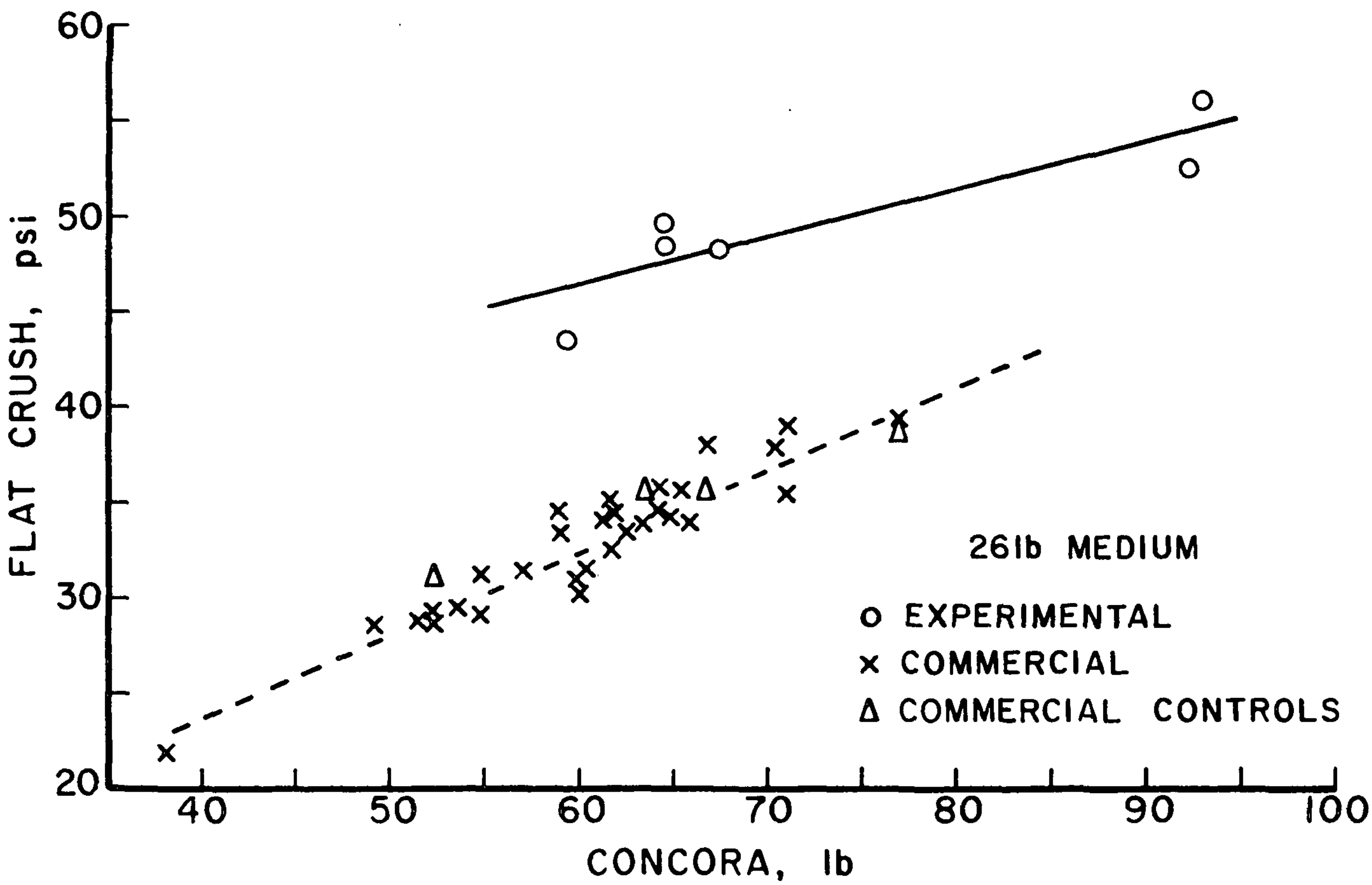

Figure 37. Flat crush vs. Concora for various mediums.

In preparing the experimental mediums, the pressing conditions were varied to increase density. Figure 38 shows that, on the average, the experimental sheets exhibited modestly higher TAPPI densities (using TAPPI caliper measurements) than the commercial controls. However, densities, based on the

IPC caliper gage (35) showed that the experimental mediums were much denser than the commercial mediums. Therefore, it would be expected that the experimental sheets would exhibit higher compressive strengths (Fig. 35), flat crush (Fig. 36), and other sheet properties (as discussed in the following text).

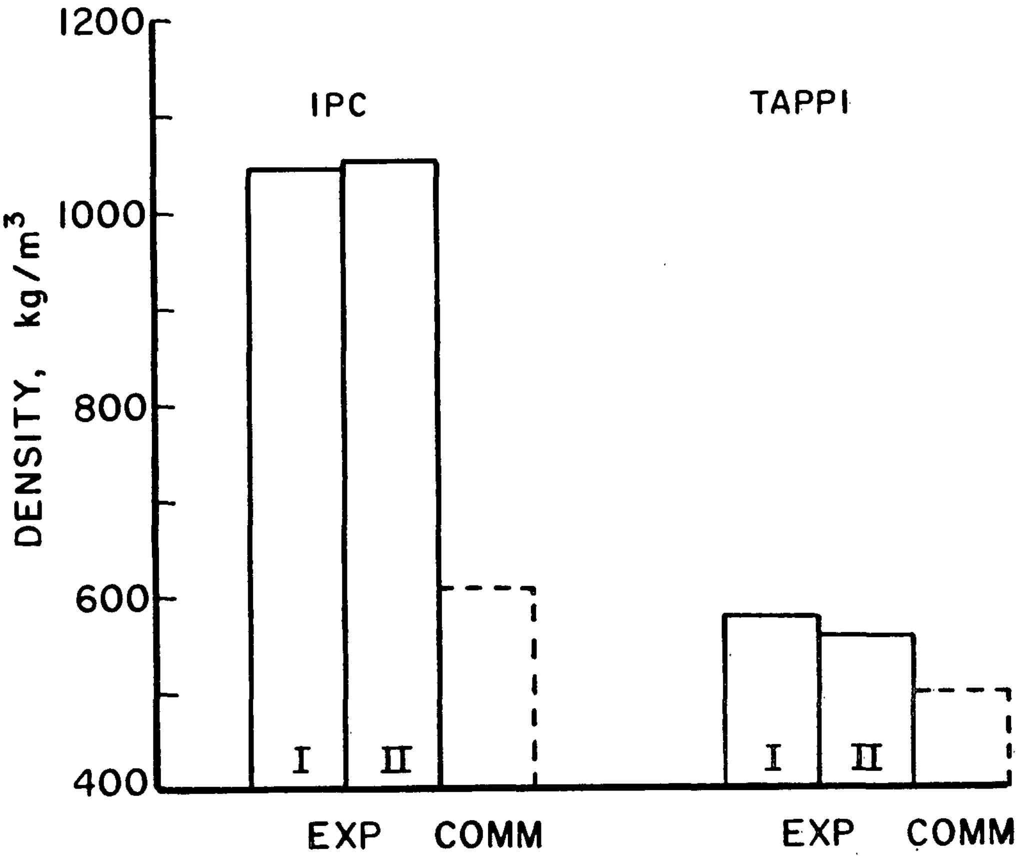

Figure 38. Densities of experimental and commercial mediums using IPC and TAPPI caliper measurements.

Figure 39 shows that the experimental sheets averaged about 20% higher MD and 33% higher CD compressive strengths than the commercial controls. The higher MD compressive strength (and other sheet properties), coupled with better forming (higher retention ratios), resulted in the 37% higher flat crush discussed above. The higher CD strengths achieved should increase the medium contribution to the ECT of combined board in a similar fashion (33).

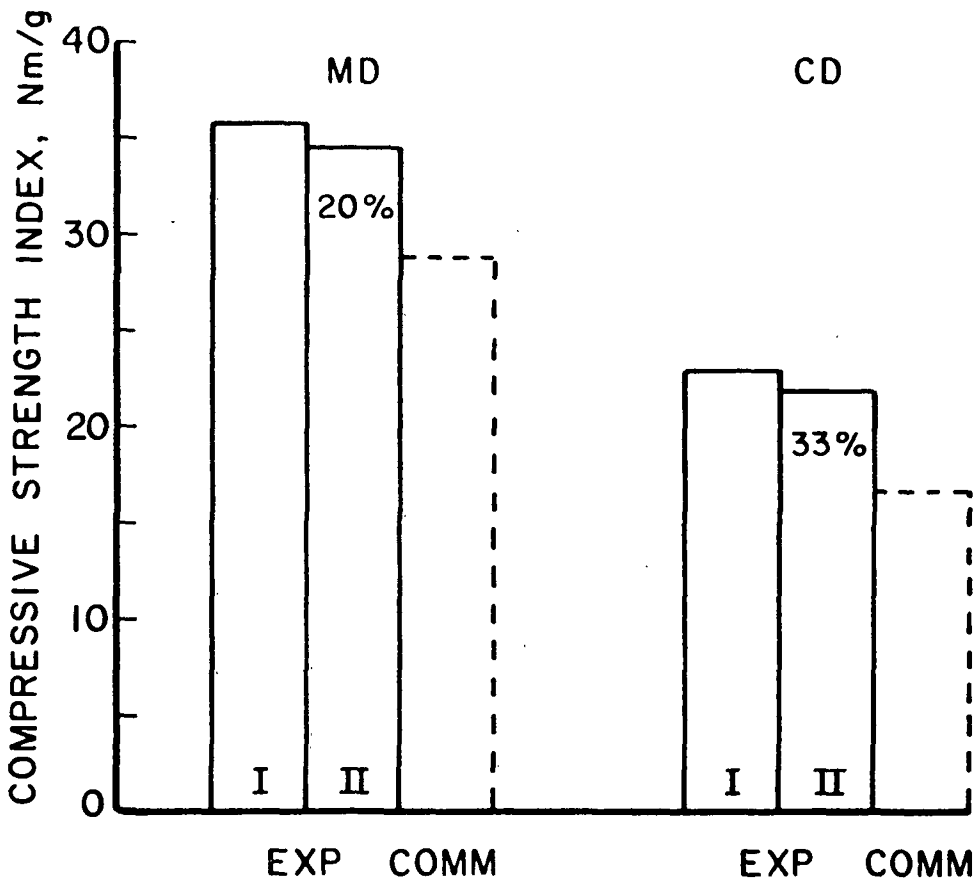

Figure 39. Compressive strengths of experimental and commercial mediums.

Figure 40 indicates that the experimental mediums exhibited higher tensile strengths in both directions due to the increased densification. The higher MD tensile strengths (68%) may help the forming because the medium can withstand higher stresses before approaching fracture.

Because of the increased densification, the in-plane specific moduli ($E_x/\rho$and E_y/ρ) of the experimental mediums were higher than the values obtained on the commercial medium controls (Fig. 41). These higher moduli contributed to the increases in compressive strength and flat crush. The transverse modulus (E_z/ρ) also effects compressive strength. A high transverse modulus (high bonding) means the fibrous layers within the sheet can better resist delamination and bond breakage under compressive stresses such as flat crush. The transverse modulus also appears to affect formability, as discussed in a later section. Figure 41 shows that the experimental mediums exhibited about a 136% increase in E_Z. This is due, in part, to the densification, but also may reflect the fact that the experimental sheets generally exhibited some shrinkage during drying. It is known that the transverse moduli are lowered substantially by wet straining before the drying process. (See Fig. 42 taken from E. Fleischman's thesis.) Thus shrinkage, the reverse of wet straining, would be expected to increase E_Z/ρ, as well as the transverse shear moduli, G_{XZ}/ρ and G_{YZ}/ρ. The latter are also known to increase compressive strength.

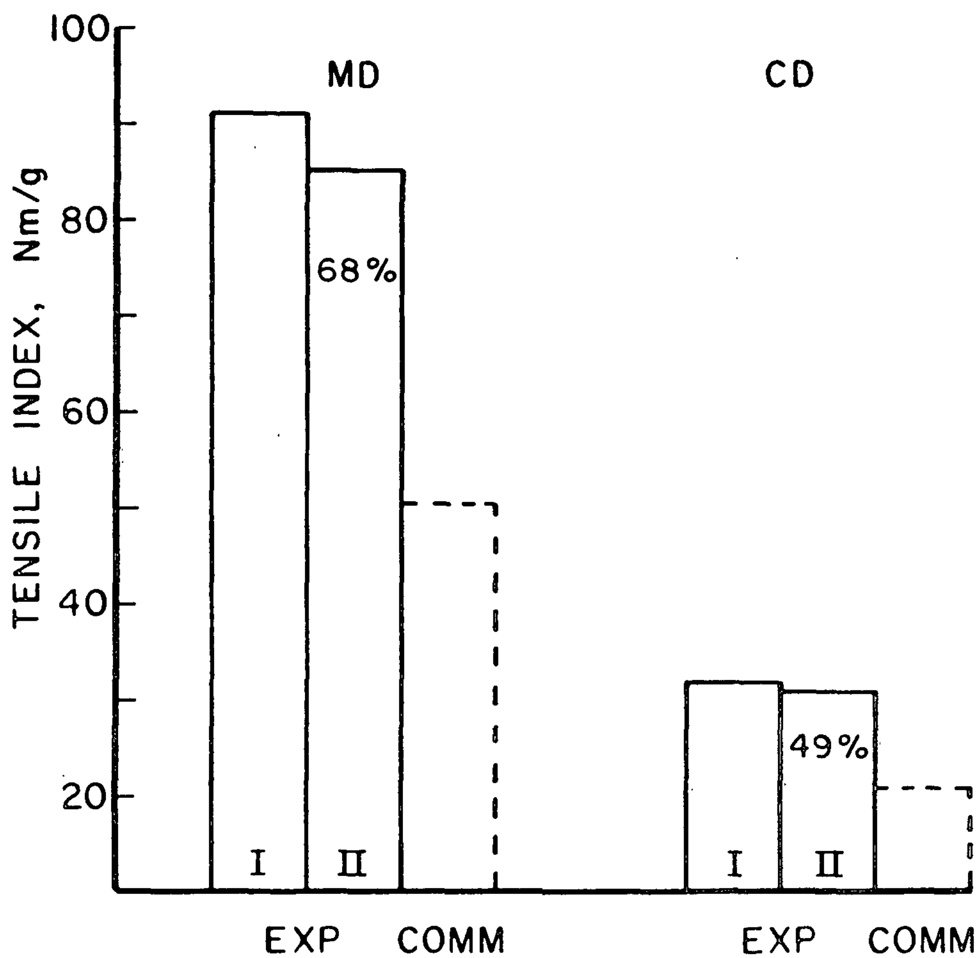

Figure 40. Tensile strengths of experimental and commercial mediums.

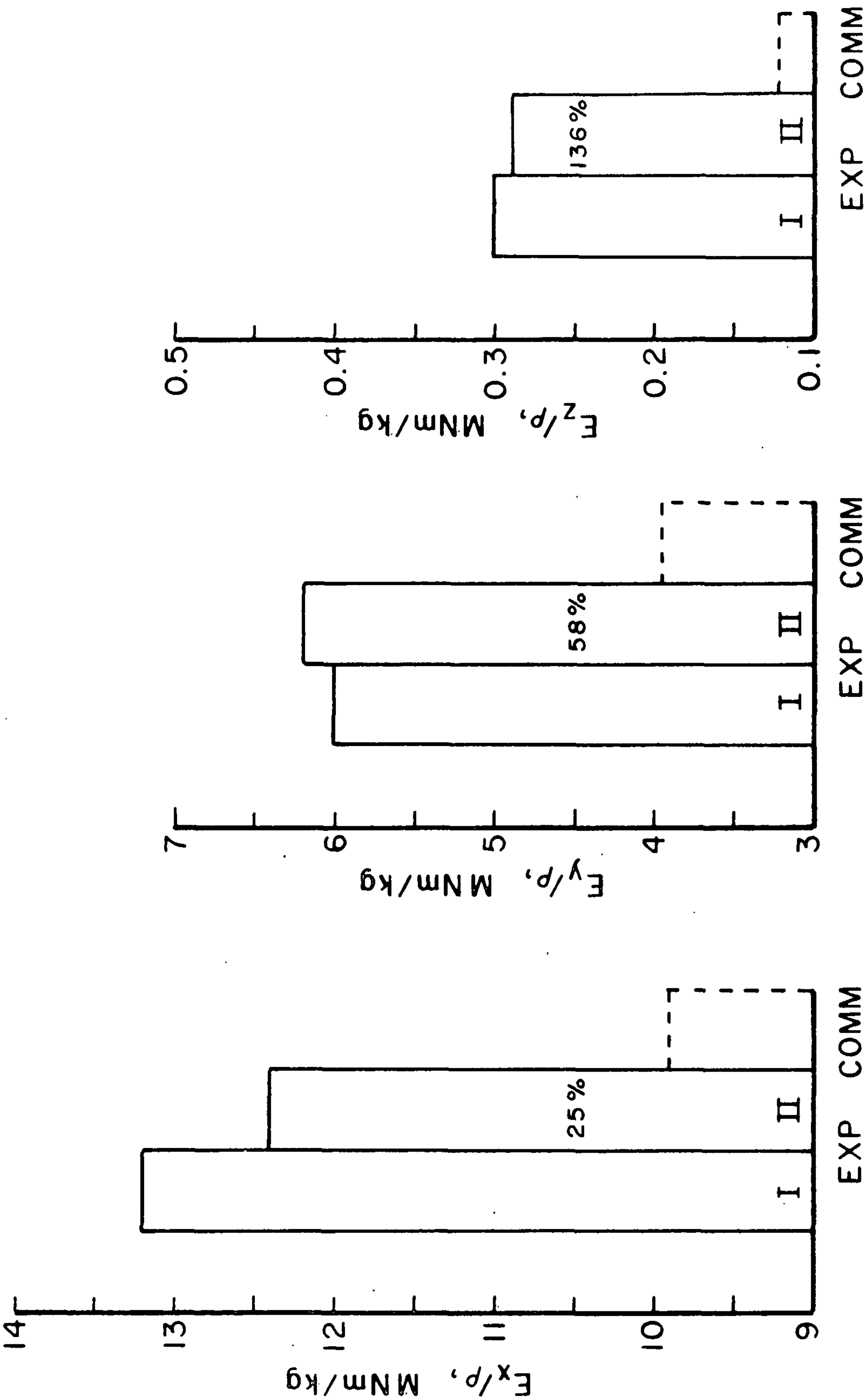

Figure 41. Specific elastic moduli of medium.

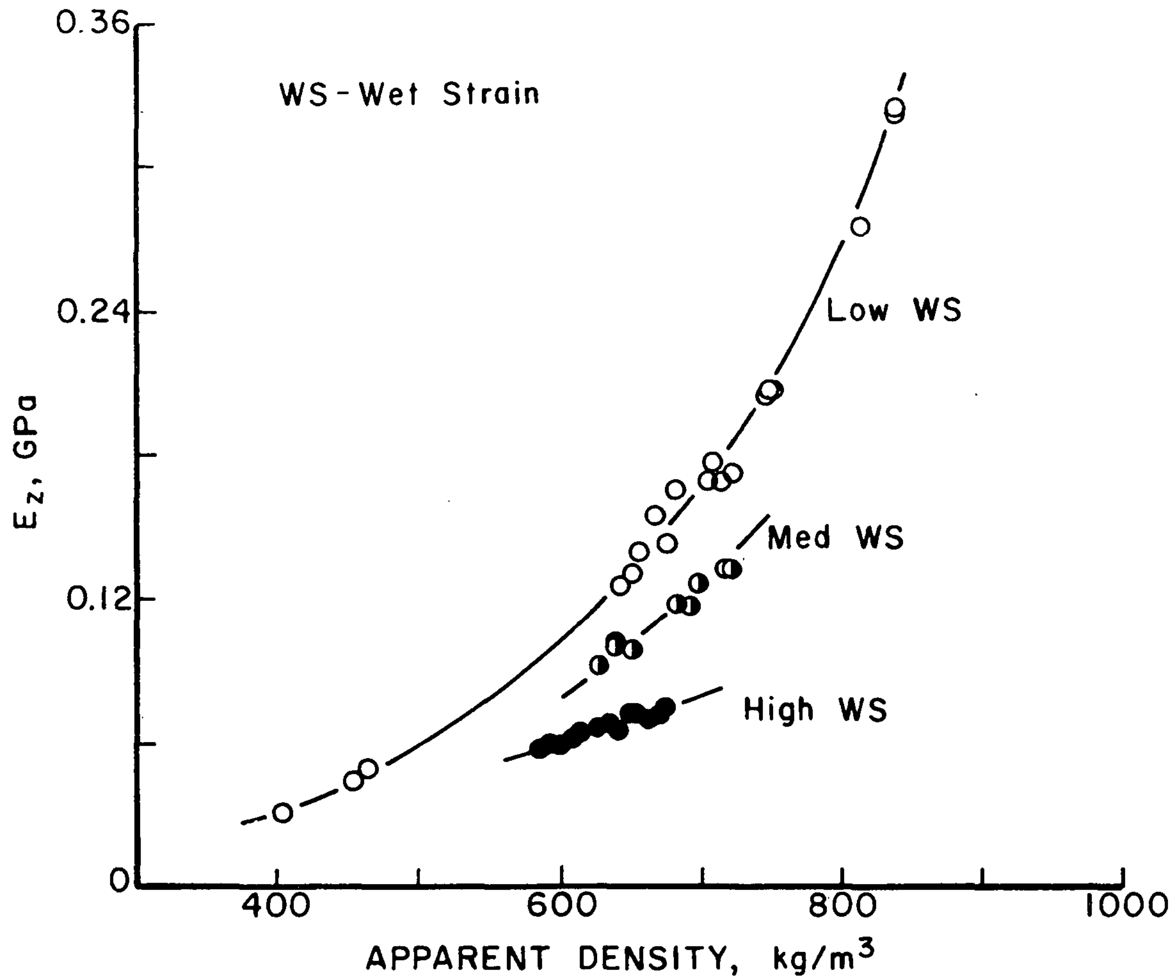

Figure 42. Wet straining markedly reduces the transverse elastic stiffness, C_{33} (almost equal to E_z).

The pressing-drying conditions used tended to result in higher than normal MD, and in some cases, CD stretch values. This would be consistent with shrinkage in the experimental sheets. Higher MD stretch should be beneficial in the corrugating operation. One of the objects of other work is to determine how the interrelated effects of densification, wet straining and fiber orientation contribute to the high compressive performances of these sheets. Increased MD wet straining should increase the MD modulus and decrease the CD modulus, MD stretch and the transverse moduli. The changes in CD modulus will, however, be affected by the degree of wet strain or shrinkage in the cross direction so the wet strains in both directions need consideration. Fiber orientation must also be considered because it, along with the wet strains, affects the properties in the two directions.

3. Flat Crush and Retention of Compressive Strength

Conceptually, flat crush strength can be expressed as the product of the base compressive strength of the medium times the fraction of the compressive strength retained after fluting (retention ratio). Thus, flat crush strength may be viewed as

Flat Crush = Base Medium Compressive Strength x Retention Ratio

It has been established that the base compressive strength (MD STFI) is dependent on the elastic stiffnesses, E_X/ρ, G_{XZ}/ρ, and E_Z/ρ (33). Two forms of this relationship are shown below:

$$\text{MD Compressive Index} = k\ (E_X/\rho)^{3/4}\quad (E_Z/\rho)^{1/4}$$
$$= k\ (E_X/\rho)^{1/2}\quad (G_{XZ}/\rho)^{1/2}$$

During forming, the bending strains induced in the medium will depend on both E_X/ρ and G_{XZ}/ρ as well as the thickness. In the fluting process, the severe bending strains are relieved, in part, by the simultaneous shear strains. High compressive strength or flat crush requires high E_X, E_Z, and/or G_{XZ}, with the restraint that if G_{XZ} becomes too high, losses may increase.

Analysis of data on commercial mediums indicates that Concora strength is dependent on both the initial MD STFI compressive strength and the transverse modulus E_Z/ρ. Conceptually, this may be viewed as the product of the base strength times a factor relating to the retention of strength in the fluting operation. Thus, a high E_Z/ρ would tend to prevent fiber bond breakage and delamination in the fluting process and, thus, promote greater retention of compressive strength.

A relationship of this type appears to explain, at least in part, the high flat crush strengths achieved with the experimental mediums made for this study (Fig. 43). From this viewpoint, densification increased both the base compressive strength and, in a substantial way, the transverse modulus (E_Z/ρ) of the experimental mediums. Taken together, they significantly increased flat crush.

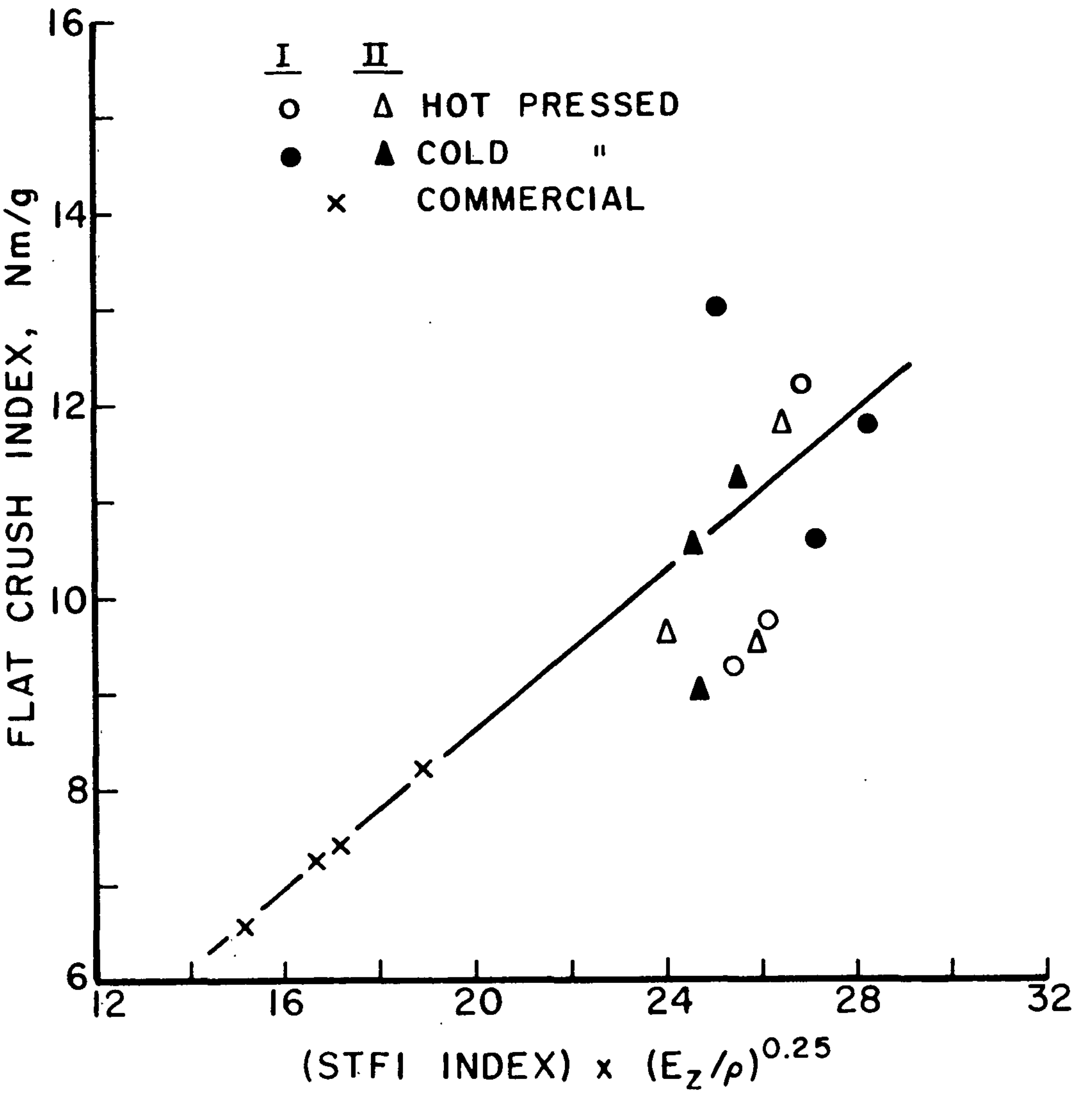

Figure 43. Relation between flat crush, base STFI strength and the transverse modulus.

An analysis of the bending and shear strains in the labyrinth, to identify the factors affecting retention, is in process. Preliminary results appear to be consistent with expectations that the transverse modulus (E_Z/ρ) is one of the main factors affecting retention.

To extend the above and assist in identifying the papermaking factors responsible for higher retention ratios and flat crush values, additional experimental sheets are being made with varying amounts of MD wet strain and densification by pressing. This requires consideration of the interacting effects of the sheet moisture at the time of pressing and straining, and the pressing and straining conditions.

In addition, work is underway to determine how the elastic properties of the sheet are degraded when medium is subjected to bending and shear strains. If the previously stated theory is correct, the losses in compressive strength during the fluting process should be related to losses in the elastic moduli. The experimental mediums are drawn over two opposing anvils to simulate the fluting labyrinth (Fig. 44) and the elastic properties before and after treatment are compared. As the medium is bent around the anvils, it is subjected to both bending and shear strains. Test conditions can be selected that are severe enough to damage the fiber-to-fiber bonding and degrade elastic properties. Preliminary results suggest that the losses in the elastic moduli parallel the changes in compressive strength.

Note: This work on the fundamentals of the fluting process and improving medium properties, initiated under the cold corrugating project to produce the results described here, is continuing under separate Institute funding.

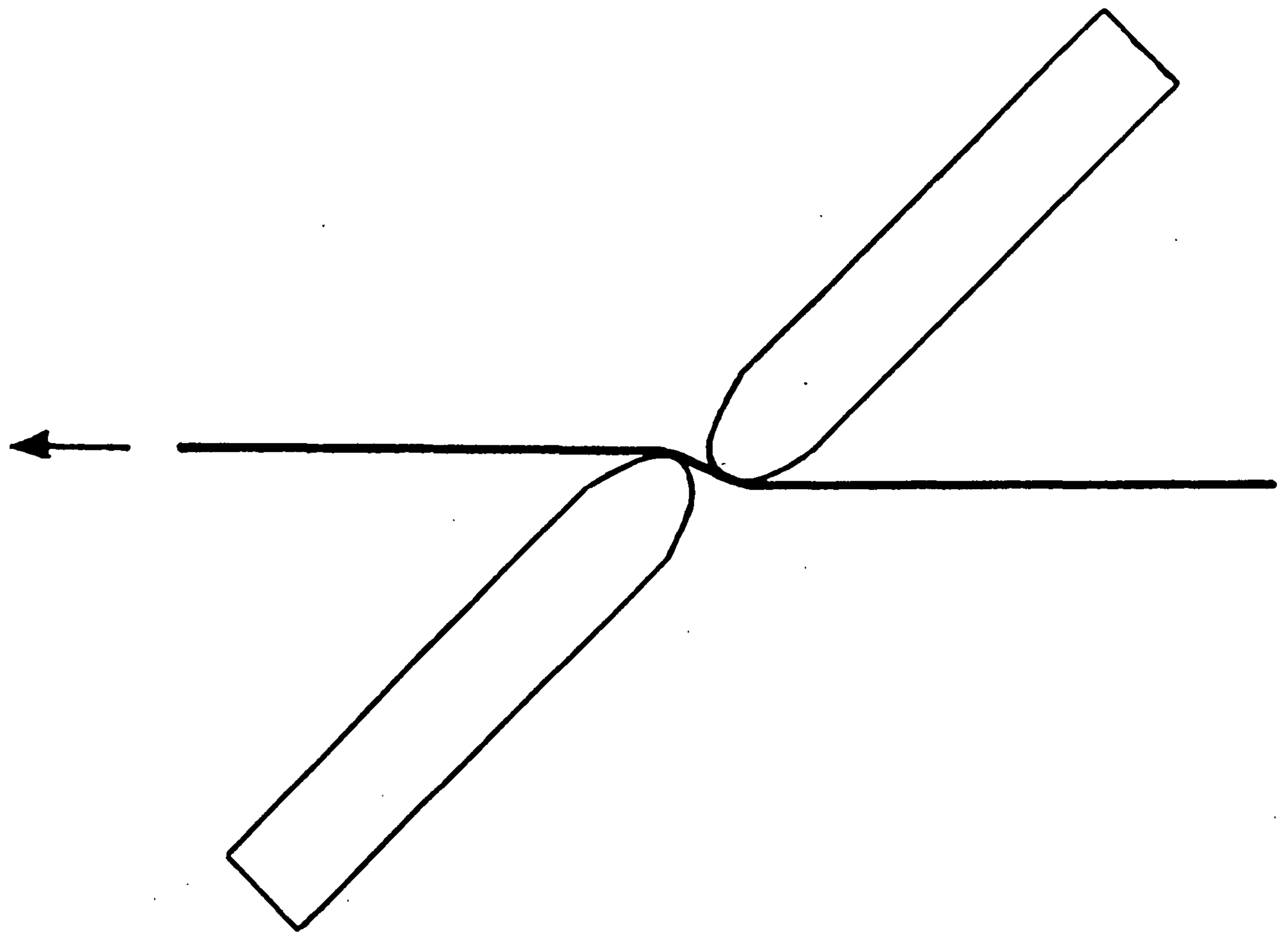

Figure 44. Schematic of apparatus for determining degradation of properties due to bending and shear.

E. COMPARATIVE EVALUATION OF COLD AND HOT CORRUGATING

The results of several exploratory and developmental experiments on fluting were presented in earlier sections. To provide a single, detailed comparative evaluation of hot and cold forming, about 35 different corrugating mediums were tested thoroughly and then converted into single face board on the laboratory single facer, by both processes. These mediums were selected to represent the wide variety of furnishes, pulping processes, wood species, and other variables that affect medium properties. While not comprehensive, the collection is believed to be representative of 26-lb/MSF mediums. In this section, we present data from testing of the uncorrugated mediums, and from the two sets of single face boards.

The laboratory single facer used for these tests is a Langston XD type of machine with a 12-inch working width on the corrugating rolls. With this width, roll crowning is unnecessary. With this exception, the machine is much like any fingered commercial machine of the XD vintage. A few extra instruments are provided for convenience of laboratory operation.

Tables XVIA and B present data on the fiber furnish and basic properties of the 35 mediums; all measurements on uncorrugated samples of the mediums were made under standard testing conditions and procedures. The fiber furnish data are the best available, but may differ somewhat from the furnishes actually used.

Medium runnability data are presented in Table XVII. For these tests, 600 fpm was taken as the maximum running speed and 3 pli as the maximum medium tension. The onset of medium fracture was used to define the limits of runnability.

Table XVIII presents comparative data from single face board made by both processes. Table XIX presents data on the effects on single face board properties of cold running with and without medium treatment agents for those mediums that could be run both ways (all other mediums fractured without pretreatment).

For all data types, mean values and coefficients of variation are given to illustrate averages and scatter. Data for some additional operating conditions were collected, but have been omitted from the tables because they are identical to the data included. These omissions are explained in the table footnotes or in the text.

TABLE XVIA

PROPERTIES OF UNCORRUGATED MEDIUM

Code	Fiber Furnish	Basis Weight, lb/MSF	TAPPI Caliper, mils	TAPPI Density, lb/ft^3	Moisture Content, %	Water Drop,[a] sec	Porosity, mL/min	Smoothness,[a] mL/min	Friction Coeff.,[a]
1	100% OCC	25.8	10.1	30.7	5.7	302	646	2867	0.34
2	30% OCC/70% NSSC	26.8	10.7	30.1	5.7	26	898	2798	0.47
3	40% Recy/60% NSSC	26.4	9.9	32.0	6.2	57	1098	2512	0.39
4	25% Recy/75% NSSC	28.3	11.0	30.9	6.7	63	663	3206	0.38
5	70% OCC/30% NSSC	26.4	11.8	26.8	5.2	23	740	3630	0.42
6	100% OCC	26.6	9.8	32.6	6.2	397	508	3142	0.35
7	100% Recy	27.2	10.0	32.6	6.1	18	1011	2546	0.27
8	42% OCC/58% gr. liq. rd adler vap. pH	25.6	10.1	30.4	6.6	600	338	2808	0.48
9	100% OCC	28.0	10.9	30.8	5.9	189	574	3057	0.40
10	15% OCC/15% clip/70% hdwd, cyld	26.6	10.6	30.1	4.8	33	488	2918	0.42
11	100% Recy	29.0	10.8	32.3	6.4	32	909	3151	0.47
12	100% OCC	25.8	9.7	31.9	5.6	431	945	2449	0.30
13	20% OCC/10% clip/70% wd pulp, crbn	26.2	9.6	32.8	4.4	139	1005	2180	0.43
14	100% Recy	26.4	10.3	30.8	4.1	162	310	3150	0.40
15	35% waste/65% gr. liq. pH, no pmy	26.2	10.9	28.8	4.5	600	445	3234	0.48
16	100% Recy	26.4	9.6	33.0	5.3	59	340	2570	0.38
17	30% Recy/10% kraft/60% NSSc	26.6	9.4	34.0	5.8	41	290	2431	0.54
18	20% Recy/80% NSSC	26.5	10.7	29.7	6.2	504	846	2759	0.51
19	15% OCC/85% Hdwd crbn	25.2	10.9	27.7	5.7	76	892	3465	0.55
20	25% Recy/75% crbn-bicrbn	25.5	10.3	29.9	5.9	30	332	2963	0.58
21	30% Recy/70% NSSC	26.0	9.7	32.2	5.7	97	958	2606	0.50
22	30% Clip/20% kraft/50% NSSC	25.9	10.1	30.8	5.3	130	522	2932	0.56
23	30% Recy/70% modf. NSSC	25.8	11.0	28.1	5.3	138	532	3352	0.56
24	30% Clip/70% NSSc	25.4	10.1	30.1	6.5	38	1435	2565	0.46
25	100% gr. liq. (NS)	25.6	9.2	33.4	6.7	93	360	2485	0.57
26	15% Clip/85% bark, nonslf cook	26.2	10.4	30.2	6.6	145	840	2800	0.57
27	NSSC + clip + screenings + sawdust pip	26.9	11.8	27.4	6.1	61	668	3458	0.56
28	100% vap. pH, twin wire	25.9	10.1	30.7	8.5	27	754	2457	0.60
29	12% OCC/13% clip/75% hdwd, four.	26.1	9.8	31.9	6.1	98	430	2674	0.46
30	100% softwood, NSSC	27.0	10.2	31.8	9.4	20	878	2149	0.58
31	80% NH_4SO_3/20% kraft clip, NSSC	25.6	10.2	30.1	7.6	55	1000	2854	0.54
32	20% clip/20% broke/60% gr. liq.	25.5	10.0	30.6	4.4	499	1166	2708	0.56
33	--	27.2	10.4	31.4	4.8	426	914	2824	0.56
34	33% kraft clip/67% NSSC, four.	27.7	9.9	33.6	5.4	122	842	2481	0.56
35	--	26.2	10.3	30.5	4.0	230	680	3054	0.55
Mean		26.4	10.29	30.9	5.87	170	722	2833	0.479
Coeff. of Variation, %		3.2	5.8	5.6	19	105	39	13	19

[a]Average of felt and wire side.

TABLE XVIB

PROPERTIES OF UNCORRUGATED MEDIUM

Code	Concora, psi	Mullen, psia.	Ring Crush, lb/in MD	Tensile, lb/in MD	Tensile, lb/in CD	Stretch, % MD	Stretch, % CD	Tensile Energy Absorption, ft-lb/ft^2 MD	Tensile Energy Absorption, ft-lb/ft^2 CD	Extensional Stiffness, lb/in MD	Extensional Stiffness, lb/in CD
1	35.3	42	5.9	36.0	14.7	1.37	2.09	3.87	2.76	4510	1880
2	39.4	42	6.8	38.1	15.0	1.16	2.51	3.33	3.34	4700	1680
3	38.9	43	6.9	39.3	14.3	1.29	2.15	3.75	2.74	4640	1710
4	38.6	51	7.6	45.0	17.5	1.53	2.24	5.32	3.50	4590	2040
5	35.9	40	5.2	34.2	14.3	1.08	3.17	2.80	4.04	4510	1340
6	36.7	46	6.6	38.4	18.6	1.39	3.18	4.20	5.32	4690	1880
7	22.9	43	5.3	34.6	14.1	1.00	2.82	2.64	3.63	4966	1608
8	32.8	42	5.5	40.5	14.8	1.39	2.23	4.49	2.99	5046	1879
9	37.0	53	6.0	45.1	16.4	1.64	2.90	5.90	4.44	5255	1932
10	37.9	42	7.0	37.9	14.4	1.40	1.87	4.19	2.36	4764	1622
11	40.0	57	6.9	53.6	17.5	1.42	4.24	5.66	6.92	6176	1791
12	31.4	53	5.7	45.5	15.5	1.66	3.12	5.93	4.48	5259	1767
13	36.0	35	5.8	33.1	13.9	1.36	2.29	3.59	2.85	4464	1806
14	37.0	48	5.2	38.6	15.5	1.33	3.41	3.98	4.79	5036	1359
15	32.1	34	5.0	30.2	12.8	1.29	1.89	3.06	2.13	4159	1781
16	34.1	53	5.3	44.0	16.5	1.46	3.50	4.92	5.37	5413	1743
17	42.2	44	7.8	47.6	15.4	1.48	2.62	5.44	3.73	5220	1810
18	38.2	40	7.7	34.9	17.0	1.22	1.79	3.22	2.58	4360	2100
19	29.6	30	5.1	27.7	12.5	1.03	1.57	2.22	1.63	3900	1610
20	31.3	27	6.0	25.8	13.1	0.94	1.30	1.74	1.37	3770	1730
21	46.1	39	7.1	43.2	14.9	1.04	2.57	3.24	3.50	5410	1810
22	35.4	46	7.7	49.6	19.0	1.20	2.97	4.64	5.09	5590	1900
23	42.3	41	7.2	39.9	13.8	1.60	2.43	5.08	3.11	4460	1690
24	32.9	35	6.7	36.1	13.6	1.09	1.94	2.93	2.53	4785	1823
25	38.3	40	6.2	37.2	14.9	1.20	1.72	3.48	2.31	4887	1956
26	37.5	32	6.4	32.7	13.5	1.26	2.24	3.27	2.44	4219	1466
27	42.6	51	7.3	49.4	18.2	1.33	3.43	4.99	5.85	5818	1854
28	36.1	32	7.0	34.6	14.5	0.98	1.32	2.50	1.57	4981	1982
29	38.5	40	6.4	39.5	14.9	1.29	1.98	3.86	2.58	4589	1724
30	39.4	47	7.3	53.3	16.2	1.28	2.36	5.02	3.20	6219	1661
31	37.0	37	6.6	38.7	16.0	1.48	2.06	4.52	2.91	4876	2018
32	35.9	40	6.7	38.8	15.7	1.37	1.84	4.15	2.54	5106	2215
33	32.2	36	6.1	31.1	14.9	1.40	2.12	3.41	2.81	4072	1905
34	31.4	41	6.4	36.0	16.3	0.96	2.30	2.56	3.39	5536	2115
35	31.0	36	6.4	28.0	15.9	1.31	2.06	2.88	2.90	3848	2088
Mean	36.1	41.6	6.42	38.8	15.3	1.29	2.41	3.91	3.36	4852	1807
Coeff. of Variation, %	12	17	13	18	10	15	27	28	38	12	11

TABLE XVII

MEDIUM RUNNABILITY RESULTS

Code	Draw Factor at 600 fpm with MTA @ 1 pli	Draw Factor at 600 fpm w/o MTA @ 3/4 pli	MTA[a] Consumption, lb/MMSF	Picking	Runnability Rating with MTA fpm-pli[b]	Runnability Rating w/o MTA fpm-pli
1	1.448	1.446	2.5	No	600-3	600-3
2	1.444	1.436	9.1	No	600-1 1/2	600-1 1/2
3	1.440	1.437	7.5	No	600-3	600-2 1/2
4	1.448	1.438	11.1	Yes	600-3	600-3
5	1.441	1.425	19.7	No	600-1 1/2	600-1 1/2
6	1.448	1.449	5.5	No	600-3	600-3
7	1.452	1.449	6.5	No	600-3	600-3
8	1.448	1.447	12.8	No	600-3	600-3
9	1.449	1.447	16.8	No	600-3	600-3
10	1.439	1.435	5.7	No	600-3	600-1/2
11	1.450	1.447	9.5	No	600-3	600-3
12	1.448	1.450	2.8	No	600-3	600-3
13	1.433	1.434	2.2	No	600-3	600-1 1/2
14	1.448	1.443	1.8	No	600-3	600-3
15	1.437	1.416	10.4	No	600-3	600-3/4
16	1.450	1.449	6.0	No	600-3	600-3
17	1.444	F	6.4	No	600-3	F
18	1.435	F	10.4	Yes	600-2 1/2	F
19	1.430	F	5.6	No	600-3	F
20	F	F	1.2	No	400-1	F
21	1.434	F	8.0	No	600-3	F
22	1.433	F	2.0	Yes	600-1 1/2	F
23	F	F	7.2	No	600-3	F
24	1.440	F	4.8	No	600-3	F
25	1.439	F	2.2	No	600-3	F
26	1.432	F	5.5	No	600-3	F
27	1.450	F	18.8	No	600-3	F
28	1.435	F	4.6	No	600-3	F
29	1.442	F	2.4	No	600-3	F
30	1.440	F	2.2	No	600-3	F
31	1.441	F	0.7	No	600-3	F
32	1.443	F	12.3	No	600-3	F
33	1.439	F	6.4	No	600-3	F
34	1.432	F	1.5	No	600-3	F
35	1.433	F	2.8	No	600-3	F
Mean	1.4409	1.4405	6.71		600 fpm & 3 pli were the maximum speed & tension used	
Coeff. of Variation, %	0.5	0.7	73			

[a]MTA = Medium treatment agent.
[b]pli = Medium tension #/lineal inch.
[F]Fracture.

1. Medium Properties

The data in Tables XVIA-B reflect the extreme variability in many medium properties. Particularly noteworthy is medium friction which has a mean of 0.48 and a coefficient of variation of 19%. As was shown previously, and will be shown again in subsequent tables, friction coefficient is a critical factor in cold forming. Moisture content, another important variable in cold forming, shows a CV of almost 30%. Attempts to relate medium properties to fiber furnish were largely unsuccessful because of the variability and uncertainty in the furnish information.

2. Runnability

Good runnability requires fracture-free forming at high speeds and high tension levels - defined as 600 fpm and 3 pli, respectively, for these trials. From the data in Table XVII, we note that in forming trials conducted without pretreatment, 16 mediums were successully formed and 19 fractured. All mediums except one* were successfully cold formed at 600 fpm when pretreatment was used. For a few mediums, fracture did occur at tension levels below 3 pli, indicating relatively poorer runnability. Such mediums should run successfully, but may be more sensitive to momentary tension spikes that occur during transient operations.

A comparison of runnability data from Table XVII and medium friction data from Table XVIA shows a strong correlation. All mediums with a friction coefficient below 0.46 were successfully cold formed without pretreatment; all those with friction coefficients above 0.48 could not be cold formed without pretreatment.

*This medium was under evaluation for fracture problems in hot corrugating and was included to provide a severe test of the cold forming process.

Draw factors, measured for operation with and without pretreatment, show little difference. A drop in the draw factor is often a good indicator of impending fracture, however.

Application rates for the pretreatment agent are highly variable and depend on medium surface properties, web tension, and applicator system loading. Only very small amounts are needed for effective function, so the application should be minimized consistent with uniformity and ease of application.

Only three of the mediums tested show signs of picking. This usually results from sticky spots or fiber bundles in the medium.

3. Single Face Property Comparisons - Cold and Hot

Table XVIII shows data from single face board made from the same components by both the hot and cold processes. All data shown were obtained from samples run at 600 fpm; properties of companion samples produced at 200 and 400 fpm show negligible differences and were omitted for clarity.

Caliper values are consistently higher for cold formed board, whereas draw factors are essentially equal to those for hot forming. As a consequence, the cold process uses about 2% less medium per unit of caliper, on the average. For comparable calipers, this translates into a 2% savings in medium cost.

Draw factors for hot and cold forming are nearly identical, and the variabilities in both draw factor and caliper are nearly the same.

In terms of structural performance, edgewise compressive strengths are comparable in value and variability. In contrast, flat crush is reduced by cold forming, by about 12% on the average. This may be due, in part, to higher caliper in the cold board, but is more likely caused by more severe or unsymmetric damage of the medium during cold forming. It is important to note that some mediums show nearly identical flat crush performance for the two forming processes, thus suggesting that medium properties may be altered to improve cold-formed flat crush performance.

Concora relates well to hot forming (r=0.90), but poorly to cold forming (r=0.56). Hence, for cold forming, a different flat crush indicator would be required.

In these tests, pin adhesion values were consistently higher from the cold process. However, adhesive application rates were not measured for either process. Hence, the significance of these results cannot be determined beyond stating that the pins were adequate to fully develop structural performance test values.

4. The Effect of Medium Treatment Agents on Properties

Sixteen of the mediums tested had friction levels sufficiently low (<0.46) to allow corrugating either with or without pretreatment. Table XIX presents performance data for these mediums for both running conditions. Included are data for caliper, draw factor, flat crush, pin adhesion, and edgewise compressive strength. Data from similar tests at 200 and 400 fpm were omitted because they do not differ from those given.

From these data, one must conclude that medium pretreatment does not affect performance, either for individual mediums or collectively. Hence, pretreatment or low friction, or both, are necessary for runnability, but not for property development.

5. Summary

Medium friction coefficient is a dominant factor in determining runnability; none of the other medium properties appears to be significant. From hot/cold forming comparisons, cold forming leads to higher caliper and lower flat crush, with all other properties being comparable. The Concora test does not give a good prediction of flat crush for cold forming. Performance variability for the two processes is comparable. Medium treatment agents have a dramatic effect on runnability, but none on properties.

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SECTION II - THE COLD CORRUGATING PROCESS ELEMENTS

PART II - BONDING

As mentioned in Part I, a corrugating process has two fundamental process elements: forming or fluting and bonding. Fluting was covered in detail in Part I; bonding will be treated in this part.

Two separate bonding processes must be considered. Single face bonding takes place in two regimes; the green bond forms under conditions of short open time and intense combining pressures for very short time intervals. The final bond forms over a longer period of time and under more gentle conditions. In contrast, the double face bonding process is characterized by relatively longer open times and low combining pressures for several seconds. Cold corrugating requires an adhesive to satisfy each or both of these processes without requiring the usual levels of heat energy for setting. At the same time, the supporting equipment must be workable within the context of commercial operation.

A. THE BONDING SYSTEM IN CORRUGATING

Bonding or bonding performance is often viewed in the narrow context of an adhesive issue but, in reality, it is much broader. Four sets of factors interact to determine bonding performance. These include the adhesive, the application of that adhesive, the components to be bonded, and the conditions under which the bond must form (the combining environment). Collectively, these four sets of factors make up the bonding system. In most bonding related developments or analysis, they should be treated in a coherent, integrated fashion as an interacting set.

1. Adhesive

a. Adhesive Constraints

Of the four elements in the bonding system, the adhesive is perhaps the most important, and the most controllable and adjustable. However, the required characteristics of the adhesive are significantly affected by the other three factors and conversely. For the development of a cold corrugating process it has been assumed (1) that the components have standard commercial properties and cannot be altered, and (2) that the combining conditions will be functionally similar to those in hot corrugating with changes limited to modest adjustments of the combining parameters. The application component of the bonding system is constrained by cost, acceptable performance in a commercial environment, and ingenuity. Developing a corrugating adhesive that sets satisfactorily without heat and within these constraints presents a difficult challenge as subsequent sections of this report will show.

b. Adhesive Requirements

i. Cost. Adhesives costs for cold corrugating must be comparable to those for the hot corrugating process. Included in the cost factor are raw materials and make-up and distribution system costs, including manpower. These cost factors, combined with the application rate required to produce satisfactory performance, determine the total adhesive cost per unit of board produced. It is this specific cost which must be controlled at competitive levels.

ii. Bonding Performance. A cold corrugating adhesive must be competitive, in every way, with conventional hot corrugating adhesives. Thus, each aspect of adhesive bonding performance must be adequate over the full commercial speed range and, collectively, these performance factors must equal or exceed those for hot corrugating. More specifically, a satisfactory cold corrugating adhesive must provide the following bonding performance features over the commercial speed range:

1. The single face green bond must be strong enough to withstand the rigors of stripping from the corrugating rolls, elevating, conveying and so on. The single face board must be free of blisters or loose back.
2. The double face bond must develop rapidly so the bond is "set" when the board arrives at the slitter-scorer. This is necessary to avoid delamination caused by the mechanical action of slitting, scoring, and cut-off. As we shall see later, fast bond development is important in avoiding a liner stretch problem, as well.
3. Final (cured and conditioned) bond strengths must be above the minimum commercial levels normally specified. A typical minimum is about 3.25 lb/lineal inch of flute (6 psi for C-flute).
4. All cured bonds must fail within one of the components, preferably the liner, with appreciable fiber tear. Bond failure within the adhesive layer is unsatisfactory; such bonds are "brittle" and fail under shock loading, although the pin adhesion levels may be adequate.
5. Application rates required to achieve the above performance characteristics must be consistent with cost requirements, warp and washboard control, and with commercially viable application equipment. Because corrugating adhesives are normally aqueous based and cold operation does not provide high thermal gradients for water removal, minimizing and balancing the application rate (actually, the amount of water added) is doubly important.

6. If at all possible, one adhesive formulation should suffice for both single and double face bonding. This would avoid the complexity of dual formulas, and make-up and distribution facilities.

All of the above adhesive performance characteristics are important. They must all be achievable over the full commercial speed range and with components from virtually all sources of all grades. Additionally, the adhesive must perform over a reasonable range of machine design and operating tolerances to avoid undue burdens on machine suppliers and on maintenance and operating personnel.

iii. Recycling. Old corrugated containers constitute an important source of recyclable fiber. The cold corrugating adhesive must not interfere with or complicate the recycling process.

iv. Make-up and Distribution System. The constraints on the adhesive make-up and distribution systems are determined by cost and the normal skill levels of the maintenance and operating personnel in a typical box plant. Although these skill levels are increasing with time, they must be recognized in selecting an adhesive and associated make-up system for cold corrugating.

v. Adhesive Handling. The adhesive selected for cold corrugating must be safe for operating personnel to work with and it must not unduly complicate or increase the clean-up and disposal tasks. Normal clean up procedures and intervals should remain workable.

vi. Water Resistant Adhesive. To produce the full complement of board grades via the cold process, it is necessary to have a satisfactory water resistant adhesive. Preferably, water resistance should be achieved by minor modifications to the basic adhesive make-up process. The water resistant version of the cold corrugating adhesive should possess all of the performance properties outlined above, plus those required to produce water resistant bonds.

2. Application

In the context of a bonding system, adhesive application is characterized by the amount of adhesive applied and the distribution of that adhesive on the flute tip. Both are extremely important to achieving the bond performance requirements of either hot or cold corrugating. Adhesive application rate and distribution are each affected by the adhesive and the application system in an interactive fashion. This must be taken into account in the selection of an adhesive and an application system.

Consistent, accurate adhesive application is perhaps the Achilles Heel of corrugating machinery. Satisfactory application is even more important and more difficult to achieve for a cold corrugating system, as the subsequent pages of this report will reveal. The problem is further complicated by migration of the adhesive after it is applied. At the single facer, migration is driven by an ejection mechanism at the pressure roll nip. In the double backer, liner slip and stretch tend to spread the adhesive and reduce its effectiveness. Both phenomena occur in both hot and cold corrugating, and both severely limit bond performance. These issues will be explored in some detail in subsequent sections on commercial prototype development and testing, but it is important to recognize these issues at this point prior to the discussion of adhesive development.

Several systems have been used for adhesive metering and application including the conventional two-roll system, gravure roll-blade combinations, injection systems (2,3), and adhesive printing systems. Each has both advantages and disadvantages, but none is totally satisfactory for either cold or hot corrugating. As subsequent parts of this report will show, a major effort was devoted to the application problem, but it remains as an issue with no fully satisfactory system yet devised.

3. Corrguating Components

Bond performance is certainly affected by the corrugating components; however, for a cold corrugating process to be competitive with the hot process, it must be workable with current commercial components. Hence, both the cold forming process and the bonding system were developed with commercial components and with the full intention that they work with all such components. While this is the only rational premise for development of a new process, there is mounting evidence that modest changes, especially in the corrugating medium, would yield good gains in performance.

4. Combining Conditions

At some time after adhesive is applied to the flute tips, the fluted medium and liner are brought together so a bond can form. This time interval is usually called the open time. The conditions to which this new joint is exposed are collectively called the combining conditions or combining environment. These conditions are dominant in controlling the quality of the resulting bond and deserve considerable attention in the development of a bonding system.

a. Single Facer Combining Conditions

i. Open Time. In a typical single facer, adhesive is applied at a point 75-90° rotational degrees ahead of the bonding nip (pressure roll-lower corrugating roll). Open time can be calculated from

$$t_o = \frac{(\frac{D}{12})(\frac{1}{2})(\frac{\theta}{360})(2\pi)(60)}{S} = \frac{\pi D\theta}{72S}$$

where

D = lower corrugating roll diameter (inches)

θ = angle from application nip to bonding nip (degrees)

S = corrugating speed (fpm)

For D = 12 inches, θ = 90°, and S = 100 and 600 fpm, t_o = 0.5 and 0.08 second, respectively. In either case, the open time is short.

ii. Combining Pressure and Time. As the medium and liner, with adhesive sandwiched between, pass through the bonding nip, they are subjected to very high pressures for a very short period of time. Typically, the pressure level is 200-300 pli. Laboratory tests have shown that the medium and liner may temporarily lose half of their caliper during this compression process. Based on these numbers, we can estimate the nip residence time as

$$t_{nrt} = \frac{3.6}{S}$$

and the average maximum pressure as

$$\bar{p}_{max} = 16 \times \text{pli}$$

For 600 fpm, the nip residence time is about 6 milliseconds; for 100 fpm, it is about 36 milliseconds. For 200 pli, $\bar{p}_{max}$ is 3200 psi.

These are only estimates of the actual conditions in the bonding nip, but they reflect the very short time intervals and the harsh conditions under which the green bond must form. These conditions are also largely responsible for driving the still fluid adhesive out of the bonding zone, resulting in severe degradation of bond quality and efficiency.

As the single face web leaves the lower corrugating roll, it is subjected to mechanical adherence and disturbing forces and other abuses which tend to separate the freshly bonded medium and liner. It is at this point that blisters normally develop. Further mechanical abuse is applied in the elevator and on the bridge, and especially as the single face web is drawn from the bridge for double backing. For short dwell time bridges - high speed single facers in close proximity to the double backer - the withdrawal process may occur long before the single face bond is fully set. The single face bond must develop in this environment and be strong enough to withstand the rigors of handling at each point in the process.

b. Double Facer Combining Conditions

i. Open Time. For double facing, open time is controlled by the separation of the glue machine and the combining point in the double backer, and machine speed. For a typical set-up, open time is given by

$$t_o = \frac{(4\ \text{ft})(60\ \text{sec/min})}{S\ \text{ft/min}}$$

At 100 fpm, t_o = 2.4 seconds and at 600 fpm, t_o = 0.4 second. These are much larger open times than those encountered at the single facer.

ii. Combining Pressure and Time. In a typical hot double backer, the combining pressure will average a small fraction of 1 psi. Peak pressures under the weight rollers may reach a few psi. In hot corrugating, pressure serves primarily to give intimate thermal contact between the lower liner and the hot plates. In cold corrugating, it plays a somewhat different role, as will be seen.

Hot plate sections vary greatly in length with machine age and production conditions, but 40-60 ft covers many of them. The hot plate section is normally followed by a pulling and cooling section that may be 20-30 ft long. Finally, there is usually some separation between the double backer and the slitter-scorer. When all these pieces are combined, the total distance available for bond formation is usually 100 ft or less. At 100 fpm, this corresponds to a bonding time of 60 seconds; at 600 fpm, the time reduces to 10 seconds.

Within the available time and conditions, the double face bond must develop enough strength to withstand the rigors of slitting, scoring, and cut-off. The demands on bond performance are increased by such factors as drag forces, liner stretch, mechanical misalignments, and so on.

In this section of the report, we have laid out the general requirements for a cold bonding system. From this, the characteristics of a suitable adhesive have been defined. The concept of a bonding system, made up of the adhesive, the application process, the components to be bonded, and the combining environment, has been stressed. However, in pursuing the development of a cold corrugating adhesive, it has been recognized that the components cannot be changed and that changes in the concepts for adhesive application and for board combining are severely restricted by machine design and retrofit issues.

B. BOND EVALUATION PROCEDURES

1. Pin Adhesion Tests

The pin adhesion tests (TAPPI Standard T821) have been the norm for the evaluation of bonds in corrugating for years. This simple test provides a direct and effective measurement of the static or slow loading rate strength of a corrugating bond. Unfortunately, the test procedures and the minimum acceptable test levels are not well-standardized within the industry. As a result, comparisons and absolute interpretations must be made with cautions. By itself, the pin test does not provide an adequate measure of bonding system performance as we shall see below. Nevertheless, the pin test is an important part of the overall bond performance evaluation set.

2. Bond Toughness or Brittleness

When bonds are subjected to more rapid loading than that used in the pin test, or to peeling loads as opposed to z-direction loads, bond failure may occur prematurely and in the adhesive film rather than in the fibrous substrates. In the field, such bonds would be referred to as "brittle" or "zipper." This important but subjective test reveals the ability of the bonds to perform under shock loads such as occur frequently in the distribution cycle. Hence, despite the fact that there is no standardized brittleness or toughness test, combined board must have the required toughness if it is to be marketable. Subjective brittleness testing is an integral part of every quality control procedure and, at least, an implied part of every order specification.

Over the course of this project several toughness tests have been evaluated; some of these will be discussed later. None of these has proven more useful or efficient than the simple locus of failure (LOF) estimate made by an experienced laboratory technician. Other tests that work at all tend to correlate well with the LOF evaluation. In the LOF test, the technician visually inspects the failed pin adhesion sample and estimates that the failure occurred in one of five zones; the adhesive (AA), the adhesive-medium interface (AC), the medium (CC), the adhesive-liner interface (AL), or in the liner (LL). For quantitative analysis, these zones are sometimes numbered from 0 to 4, in order of increasing preference. Failure within the liner (LL or 4) is always the objective. Bonds which fail consistently within the liner will satisfy the field toughness requirements. Bonds may have high pin levels yet fail within the adhesive. Such bonds would be unsatisfactory in the field.

3. Adhesive Consumption

Good bond performance is necessary, but it must be achieved with an acceptable amount of adhesive. For a cold process using an aqueous-based adhesive, more adhesive means more water which cannot be removed. This increases cost, warp, washboarding, and moisture content off the machine. The latter may adversely affect the quality of the sheeting operations and subsequent handling and finishing operations. Hence, the amount of adhesive used is an integral part of a bond performance assessment.

Measuring adhesive application rates while the machine is running under steady-state conditions is fairly straightforward (1,4). Under the short-term running conditions of testing, it is virtually impossible. Laboratory tests on finished board are satisfactory but expensive and time consuming. As a consequence, actual adhesive application rates are often unknown. Application system settings are randomly related to local application rates, so they cannot be used to give even a crude estimate of application rate. Our inability to know the adhesive application rate and to reliably control it have been very costly in this project; they are in day-to-day production, as well.

4. Adhesive Distribution Tests

While it is not an inherent bond performance feature, the distribution of the adhesive within and around the bond zone is critical to bond performance.

Distributions in the machine direction are best observed by sectioning, staining, and photomicrographing a bond cross section. Photomicrographs do not yield quantitative information but are very useful in observing the small-scale details of adhesive distribution and bond character.

Adhesive distribution in the z-direction - penetration of the components - can be easily detected by taper grinding and staining. For this test, bonded components are separated and attached to metal strips with two-sided tape. A grinding wheel is used to remove progressively more material along the length of the strip. Staining will then reveal the extent of the distribution at each depth within the component and, of course, the maximum depth of penetration.

5. Testing of Water Resistant Bonds

In order to test the strength of water resistant bonds a special fixture and test procedure were devised. For this test, a sample of board 2 inches wide and 10 flutes in length is used. Extensions on the opposite liners are used to clamp the sample so it may be subjected to a machine direction shear load. To avoid handling the samples while they are wet, a special fixture for holding six samples was built (Fig. 1). Samples are clamped in the fixture in the dry state and the complete fixture is then immersed in water for 24 hours; after soaking, the samples are drained, the fixture clamped in the Instron, and the samples tested one at a time.

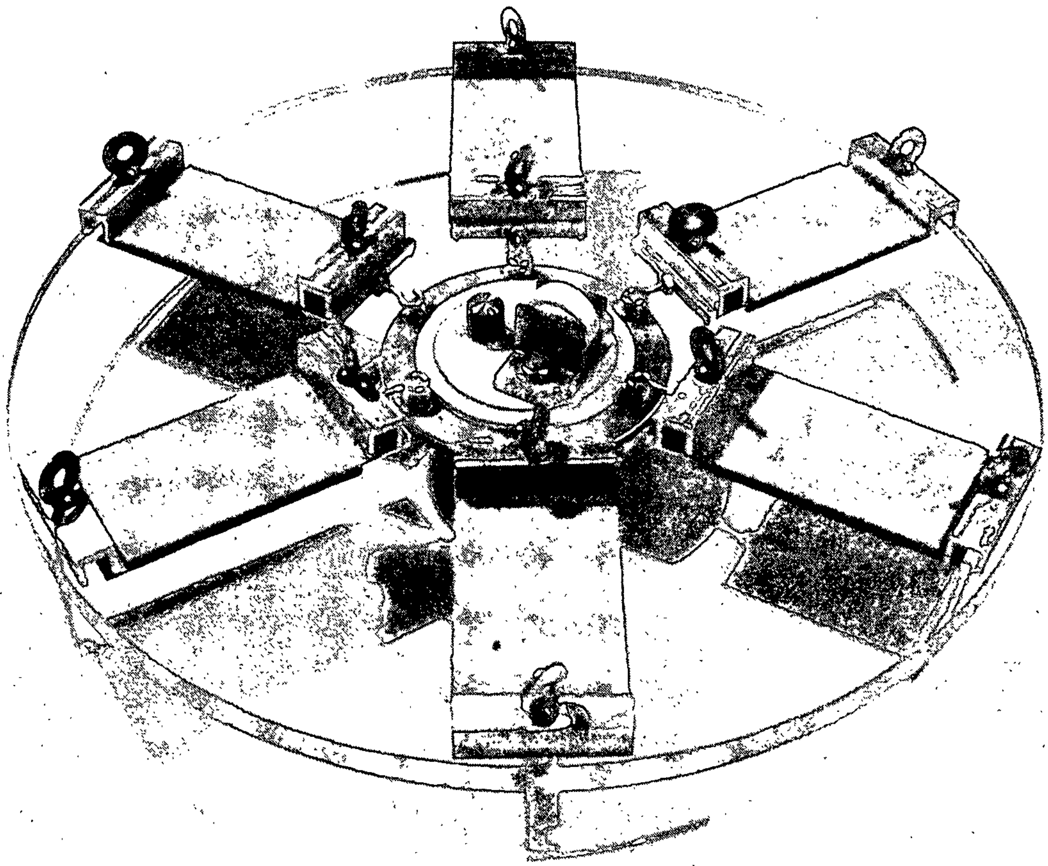


Figure 1. Wet strength test fixture.

C. COLD CORRUGATING ADHESIVE ALTERNATIVES

The basic objective of this project was to develop a successful cold corrugating process. This, in turn, required development of successful forming and bonding process elements. The foregoing sections of this part of the report have presented the requirements of the bonding system and the test procedures used to evaluate bond performance. We now turn our attention specifically to the adhesive development question.

In this section, we want to explore briefly some of the alternative types of cold-setting adhesives. This issue was reopened at various times through the project, as bonding difficulties seemed insurmountable, but always with the same result; a decision to continue with pearl starch-based adhesives.

Traditionally, water-based adhesives have been utilized in the production of corrugated board. This has been due, in part, to the state-of-the-art and to the higher cost of the alternatives. These water-based adhesives are able to make the transition from the fluid condition needed for application to the functioning bonding agent through several mechansims which produce rapid changes in viscosity. In the case of sodium silicate solutions which are often applied in the range of 30-35% solids, the bond is formed through loss of water to the board components. Sodium silicate has many desirable characteristics, among which are increased rigidity, will develop adhesion without heat, uniform, etc. However, its undesirable characteristics such as adhesion to hot-melt surface, sensitivity to high humidity, high alkalinity, lack of water resistance, etc., now limit the use of sodium silicate to a small fraction of the corrugating adhesive market.

Starch-based adhesives have been most successful in replacing silicate and, in particular, one formulation has become the standard of the industry. This formulation consists of raw unmodified cornstarch suspended in a solution of sodium hydroxide, borax, and dispersed starch. The dispersed starch or primary acts to provide adhesion as well as maintain the raw starch particles in suspension and to provide the necessary flow properties needed for smooth application. Two stages are involved in the functional bond with this adhesive. The initial or green bond is formed when sufficient heat penetrates to the adhesive to cause the raw starch to swell and imbibe water, thereby forming a gel of sufficient strength to hold the structure together. This bond is said to form within 0.1 to 10 seconds, depending upon heat transfer. The final bond strength depends upon removal of the water from the gel into the fiber structure and out of the board, if possible. Several measures have been taken with some success to retain the water in the starch adhesive to strengthen the green and dry bonds. Jet cooking procedures and the "no carrier" formulations are included among these.

Conventional corrugating adhesives are gelatinized by heat to produce an initial or green bond. In cold corrugating, heat is not available so adhesion must be developed by physical or chemical changes in the adhesive, induced by other driving forces. Hot melts and adhesives with temperature-sensitive viscosities set by increases in viscosity on cooling. Tack can also result from a change in the chemistry of the adhesive, such as setback or cross-linking. Loss of solvent is the dominant effect in bond formation by aqueous dispersed adhesives (8). With water-based adhesives the ultimate bond strength will develop upon complete drying of the adhesive as it does in conventional corrugating.

Hot melt adhesives have been used in some corrugating applications but are regarded as too expensive for general use. Other, aqueous based, cold-set adhesives have been developed. Some, like Borden's modified polyvinyl alcohol (9) or Dural's P-1212 (10) are based on synthetic polymers. These are also expensive and some are not compatible with conventional starch adhesives. Other cold-setting adhesives are based on natural polymers. National Starch and Chemical patented an adhesive based on a highly modified dextrin (11). Although the adhesive showed promised, National abandoned efforts in this area, reportedly because of unavailability of suitable corrugating equipment (12).

D. EARLY ADHESIVE DEVELOPMENT

The approach of the present program proposes that a thoroughly cooked starch should be a stronger adhesive than a partially cooked starch. Further, a cooked starch adhesive that gels at a relatively high temperature, applied at a temperature above the gel point, should form a green bond upon cooling and a final bond by vapor diffusion away from the bonded area. To produce an adhesive with this property, consideration was given to a combination of modifying chemicals which are known to reduce the viscosity of the hot cooked starch and to enhance the set-back of the paste. The term "set-back" refers to the transition from a slowly increasing viscosity as the fluid cools to an abrupt elevation due to increased intermolecular association preceding gel formation. Two treatments are known to increase the set-back of modified starch over that of the unmodified product. These are acid modification of the raw starch granules and persulfate treatment as employed in the thermomechanical conversion process. Addition of certain salts such as sodium bisulfite or sodium sulfite along with ammonium persulfate to the starch as it is heated in a jet cooker raises the gelation temperature of the cooked paste. Hence, the result is a cold-set adhesive which should form a gel when brought into contact with the cooler surface of the liner.* In this manner, an instantaneous green bond should be formed which would not be dependent upon the transfer of heat through the liner. The composite of liner and medium would be used as a heat sink to solidify the adhesive, which would then dry by diffusion of moisture into the board structure. Since the functionality of a cold-set adhesive does not depend upon heat transfer in

*Early in the project, cooling was believed to be a dominant factor in green bond development. Later work showed water loss to be much more important.

short time intervals, an adhesive of this type should theoretically provide an advantage at high corrugating speeds and/or with heavyweight liner.

In work at The Institute of Paper Chemistry a starch adhesive based on thermochemical conversion at the plant site was given primary consideration as the adhesive for cold corrugating for a number of reasons. Starch is inexpensive. It is a material familiar to corrugated board plants, compatible with their current practices, and readily available. The processes for in-plant conversion are well established and well known, at least in the paper industry. Such adhesives create no new problems in recycling old containers and waste.

A patent (7) assigned to National Starch and Chemical Company describes starch-based adhesives which form a green bond by cooling. However, the examples given refer to starch dextrins and to adhesive solids contents in the range of 32-60%. The work described herein utilizes less expensive pearl cornstarch at solids contents under 39%.

Project 2696-11, started about 1972, was initiated at The Institute of Paper Chemistry for the purpose of developing a cold-set adhesive for corrugated board using starch as the basic raw material. The cold-set adhesive concept considered herein is similar in application to conventional hot-melt adhesives in that both are applied at an elevated temperature and form a bond by cooling. On the other hand, it differs in that the components of hot-melts normally are not dispersible in water, whereas the starch is.

The study was carried out in two parts. Part One consists of a study of the effect of various starch modifiers on the "adhesive" characteristics of starch. Part Two involves a study of the effect of adhesive solids content on bonding strength under several conditions of viscosity and gelation temperature.

1. Phase I - Development 1972-1973

a. Introduction

A study was made of cold-set starch corrugating adhesives comprised of pearl cornstarch, ammonium persulfate, a salt such as sodium bisulfite or sulfite, and an alkaline material. Ammonium persulfate modified the starch to permit the use of higher adhesive solids contents and the bisulfite or sulfite was used to increase the gelation temperature of the adhesive to temperatures as high as 77°C (171°F). Alkaline agents such as caustic soda, sodium bicarbonate, and sodium carbonate were added for pH control. The adhesive was prepared by passing a slurry of the starch and chemicals through a jet cooker (Fig. 2) at temperatures ranging from 230 to 280°F. Dwell time in this cooker was about 6 seconds. The solids content of the cooked adhesives ranged from approximately 20-35%. When applied to board in the corrugating operation, the adhesive sets upon cooling and drying and thus forms a bond between the liner and medium.

b. Effect of Process Variables

An exploratory study was conducted to determine the effect of several process variables on the starch adhesive properties. Increasing the bisulfite content of the starch slurry at constant persulfate level increased the viscosity and gelation* temperature of the jet-cooked product (Fig. 3), whereas the post addition of sodium sulfite or caustic soda to the cooked adhesive had little effect on viscosity and gelation temperature. The post addition of borax

*Cooked starches of this type do not "gel" in the conventional sense. However, they do increase rapidly in viscosity with decreasing temperature. Such temperature-viscosity curves can be measured conveniently with the Brabender Amylograph Viscograph from which viscosity is expressed in Brabender units (BU). In this early work "gelation temperature" was arbitrarily defined as the temperature at which the adhesive viscosity increased by 500 BU, starting from a temperature of 95°C and a spindle speed of 190 rpm. This term was later dropped as lacking special significance.

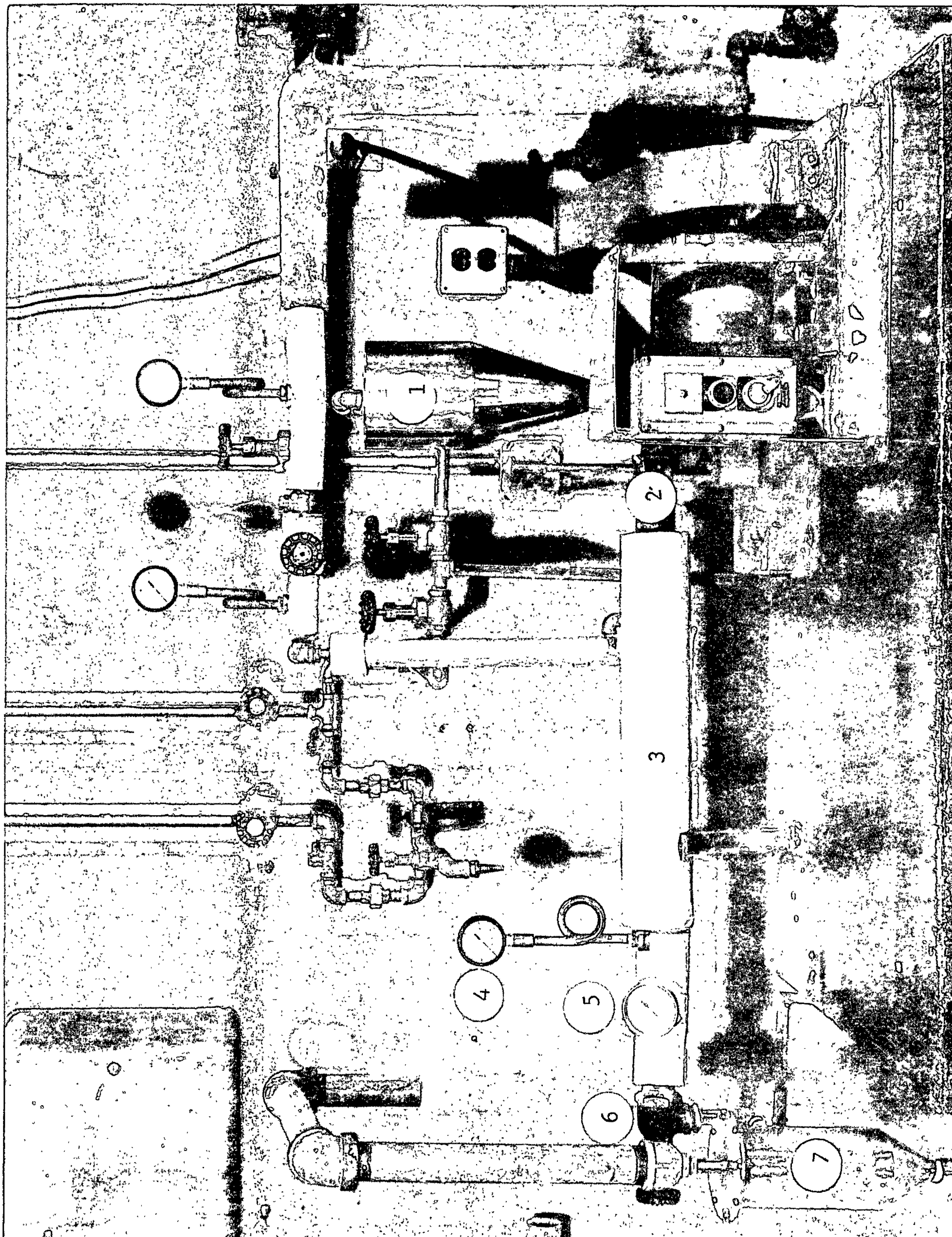


Figure 2. Jet cooker. 1. Hopper; 2. Moyno pump; 3. Jet reaction chamber; 4. Pressure indicator; 5. Temperature indicator; 6. Throttling valve; 7. Separator.

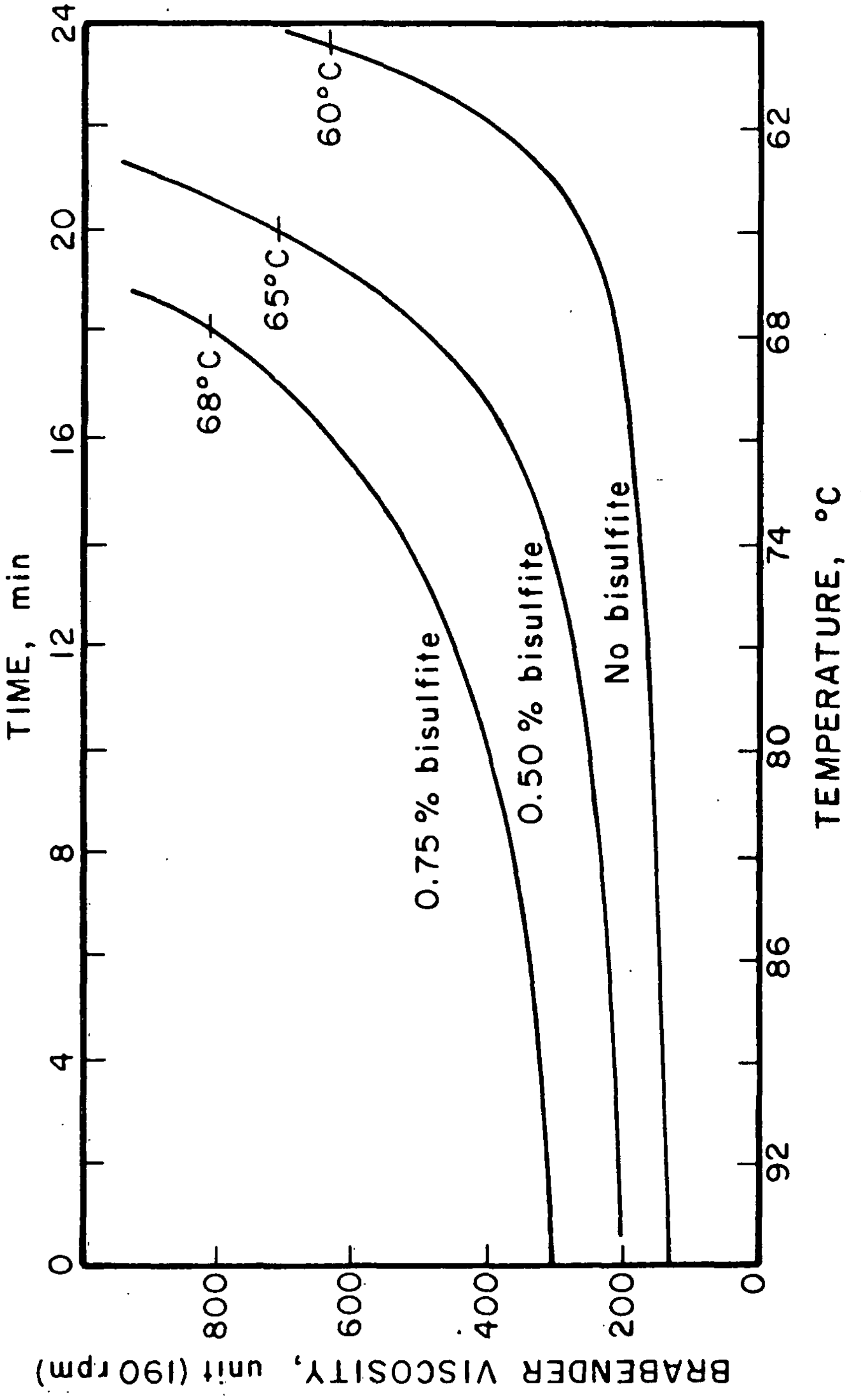


Figure 3. Viscosity vs. temperature relationships (0.5% P and 0.3% NaOH).

to a cooked adhesive containing persulfate and sulfite tended to reduce the gelation temperature. Reheating the gelled adhesive produced a material of greatly increased viscosity compared to the original cooked adhesive (Table I), thereby making the reheated product unsuitable for corrugating.

TABLE I

THE EFFECT OF REHEATING THE GELLED ADHESIVE

Sodium Sulfite, %	pH of Cooked Adhesive	Solids Content, %	Brabender Viscosity at 95°C, units at 190 rpm		Gelation Temp. °C at 190 rpm
			Initially	Reheating	
0.6	3.3	22.3	550	1080	72 (162°F)
0.6	6.3[a]	--	520	950	70 (158°F)
0.5	7.6[a]	21.6	180	1340	67 (153°F)

[a]Sodium hydroxide added.

The cold-set starch was subsequently utilized as the corrugating adhesive for a standard 26-lb medium and 42-lb liner combination under several conditions of persulfate:sulfite ratio, solids content, pH, and corrugator operating temperatures, speeds, and clearances. The adhesive metering roll clearance was set at 0.010 or 0.012 inch. The pan temperature varied from 162 to 187°F and the glue roll from 162 to 190°F over the course of the corrugator trials. In most trials, the corrugating rolls were operated at normal hot temperatures and the pressure roll was either at ambient temperature (≅80°F) or at 330°F. Corrugating speeds ranged from idle to 400 fpm. For control, a single run was made with a conventional carrier starch adhesive under typical hot operating conditions. The corrugated board was subsequently conditioned at 73°F and 50% RH and tested for pin adhesion. Representative data are given in Table II.

TABLE II

CORRUGATING TRIAL DATA

Ammonium Persulfate, %	Sodium Sulfite, %	Cooking Temp., °F	Solids, %	pH	Gelation Temp., °C	Operating Temp., °F Pan	Operating Temp., °F Glue Roll	Operating Temp., °F Press Roll	Speed, fpm	Pin Adhesion, lb	LOF[a]
0.75	0.75	230	26.3	7.6	73	184	185	Cold	Idle	87	AA or AL
									400	--[b]	
						165	166	Cold	Idle	70	AA
									400	--[b]	
0.5	0.5	250	28.8	7.5	72	187	187	Cold	Idle	80	AC
									400	--[b]	
						187	187	21C	Idle	59	AC
									400	--[b]	
0.5	0.5	250	--	6.8	--	186	186	Cold	Idle	91	LL
									100	91	AA
									200	82	AA
									400	77	AA
					(Increased press roll pressure)				Idle	89	AC
									100	84	AC or AA
									200	73	AC
									400	60	AC
						186	86	330	Idle	25	AA
									100	72	AC
									200	61	AC
									400	52	AC
		Conventional Carrier Adhesive			--	95	100	330	Idle	91	AL
									100	85	AL
									200	85	AL
									300	73	AL

[a]LOF = Locus of failure. AA = adhesive, AC = adhesive-medium interface, CC = medium
AC = adhesive-liner interface, LL = liner.

[b]The dried bond was too brittle to test for pin adhesion.

Within the limits of the experiments, the persulfate:sulfite ratio and pH had little effect on pin adhesion. Heating the corrugator pressure roll tended to reduce pin adhesion at the lowest corrugating speed. Increasing the pressure on the pressure roll appeared to have a beneficial effect on adhesion but the results were too limited to be conclusive. While the cold-set adhesive was found to provide acceptable "green" bond in most cases, the "dry" bond tended to be inadequate due largely to cohesive failure. However, the pin adhesion values in several cases approached those of the conventional adhesive used as a control, and the program was extended on this basis.

In a follow-up study, consideration was given to the effects of adhesive solids content on bond strength under several conditions of viscosity and gelation temperature. Because chemical modification of starch tends to reduce the unit bond strength, it was reasoned that increased adhesive solids would improve bonding. After examining further the process variables related to viscosity and gelation temperature (Fig. 3 and 4), a series of corrugator trials was conducted in which the starch adhesive solids content ranged from 22 to 33%; the viscosity from 70 to 420 Brabender units; and the gelation temperature from 59 to 70°C (138-158°F). The corrugator was operated with hot corrugating rolls at speeds up to 400 fpm under conditions of constant glue roll clearance and temperature but with variation in the pressure roll temperature and pressure. A steam injection-water bath was utilized as a starch-holding tank during this series in an effort to provide better uniformity and to prevent viscosity buildup which is believed to have occurred in the earlier trials.

Pin adhesion values comparable to those obtain with a conventional adhesive control were attained at a solids level of 23% and the values increased somewhat at optimum viscosity as the adhesive solids increased to 33%. Within a given narrow solids range, pin adhesion was lowest at the highest viscosity. The maximum adhesion occurred at intermediate viscosity and gelation temperatures. Hence, the cold-set starch formulation was found to provide adhesion values comparable to the conventional adhesive without the need of resorting to unusually high solids levels.

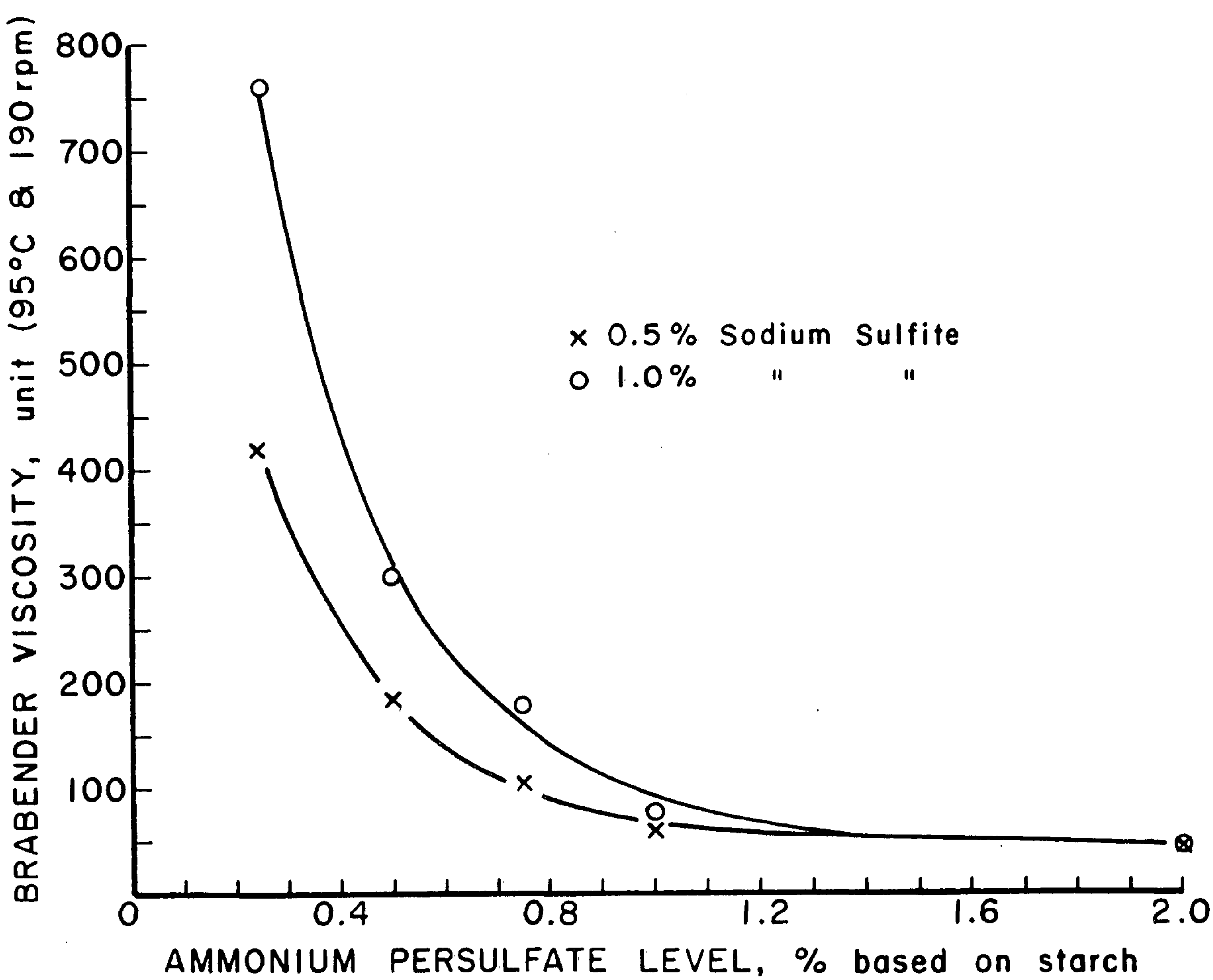


Figure 4. The effect of persulfate level on viscosity at constant adhesive solids (23-24%).

Subsequent to this base-line study, a second series of trials was conducted in which several variations of the cold-set adhesive, including one containing 2% latex on starch, were tested at approximately 23% solids at corrugating speeds up to 660 fpm (Fig. 5). The cold-set adhesive provided rather consistent pin adhesion values (≈ 70-80 lb/8 inch^2) at 23% solids at all corrugating speeds, whereas the adhesion level obtained with the carrier adhesive (21% solids) declined rapidly with increasing speed above 300-400 fpm (Fig. 6). Limited preheater capacity on the laboratory corrugator may have contributed to this decline, but some dropoff is normal with conventional adhesives at high corrugating speeds. At most, slight improvements were noted with the adhesive containing latex. Hence, the cold-set adhesive was indicated to provide an advantage over the conventional adhesive at only slightly higher solids. The results of this program were considered sufficiently encouraging to warrant continued study of starch cold-set adhesives.

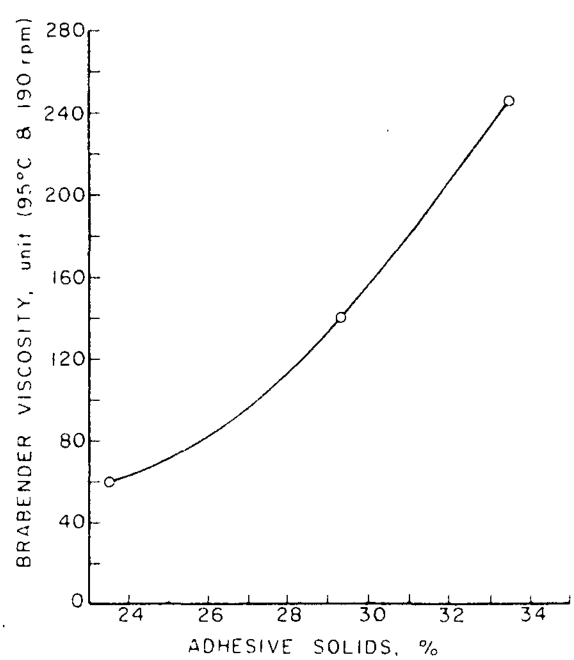


Figure 5. Viscosity as a function of adhesive solids at constant persulfate and sulfite levels (1.0% persulfate; 0.5% sulfite).

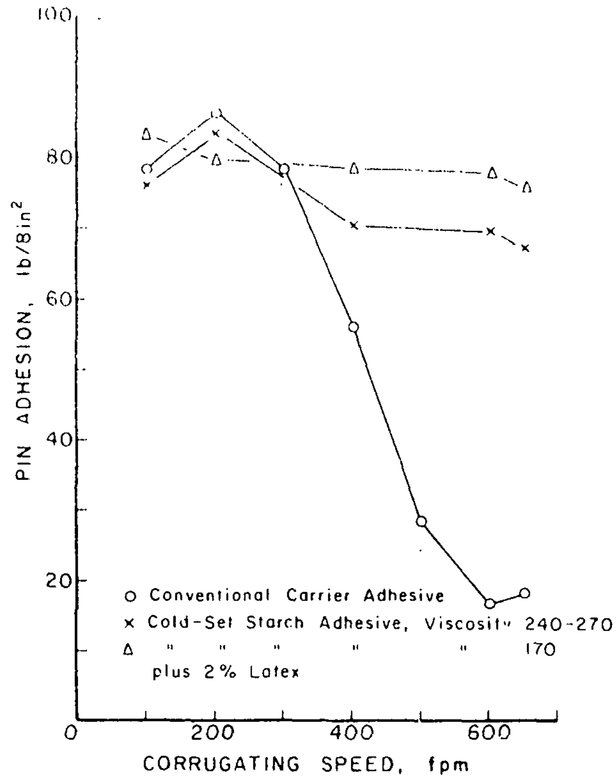


Figure 6. A comparison of cold-set and regular starch corrugating adhesives at 21-23% solids (regular medium and liner).

2. Phase 2 - Development 1973-1975

a. Introduction

In general, the results obtained in Phase 1 of the investigation of cold-set starch adhesives were very encouraging. Pin adhesion values comparable to the standard two-component starch adhesive were attained under essentially neutral conditions at a solids level of 23% and the values increased significantly as the adhesive solids increased to 33%. As the corrugating speed increased to 660 fpm the cold-set adhesive provided rather constant pin adhesion values, whereas the adhesion level for the conventional starch heat-set adhesive declined at speeds in excess of 300-400 fpm. However, a number of potential problem areas appeared in this work which suggested the need for further study.

One persistent problem was the lack of visual fiber pull in the pin adhesion test in spite of adequate adhesive strength in terms of force. In general, failure occurred at the medium-adhesive interface, suggesting a lack of penetration prior to setting of the adhesive. The second and somewhat related problem was a tendency for the cold-set adhesive to be brittle, i.e., the adhesive tended to lack flexibility after setting up. Phase 2 was directed at resolving these problem areas or, in effect, at optimizing the cold-set adhesive. In pursuing the experimental program, consideration was also given to better definition of the limitations of the process with respect to temperature and time variations as they relate to the viscosity and gelation temperature of the adhesive and, ultimately, to adhesive performance.

b. The Effect of Medium Receptivity

The effect of the receptivity of the medium on adhesive performance was explored in several series of corrugator trials in which the chemical composition of the adhesive was varied. The purpose of this work was to promote penetration of the adhesive into the board components so as to enhance fiber failure in the pin adhesion test. The first series of trials utilized a cold-set starch adhesive containing 0.5% of persulfate, 0.5% of sodium sulfite, and 0.3% of NaOH (based on starch). Two adhesive samples with this composition were prepared with different viscosities and gelation temperature. The adhesives were applied to mediums known to vary in water drop value from approximately 20-560 sec. For reference, a conventional carrier adhesive was also used. Typical results from these trials are recorded in Table III.

A subsequent series of corrugator trials utilized cold-set adhesives containing 0.5% of persulfate and 0.5% of sulfite with the following modifications.

TABLE III

THE EFFECT OF MEDIUM PROPERTIES AND ADHESIVE VISCOSITY ON PIN ADHESION

Receptivity of Medium, water drop, sec	Adhesive No.	Adhesive Solids Content, %	Final, pH	Brabender Viscosity at 95°C	Gelation Temp., °C	Operating Temperature, °F Pan	Glue Roll	Corrugating Speed, %	Pin Adhesion, lb	LOF
19-27	1	22.9	7.5	270	67(153°F)	194	205	200	56.2	CC
								400	56.6	CC or AC
								600	Medium fractured	
412-564	1	22.9	7.5	270	67(153°F)	190	205	200	64.0	AL
								400	61.2	AL
90	1	22.9	7.5	270	67(153°F)	190	205	200	65.8	AC
								400	49.0	AC
								600	43.2	AC
115-165	1	22.9	7.5	270	67(153°F)	190	205	200	75.4	AC
								400	68.8	AC
								600	59.3	AC or AL
19-27	2	22.2	7.1	165	64(147°F)	192	208	200	77.4	CC or AC
								400	63.4	CC
412-564	2	22.2	7.1	165	64(147°F)	192	210	200	75.6	AL
								400	Medium fractured	
90	2	22.2	7.1	165	64(147°F)	192	205	200	69.4	CC or AC
								400	54.6	AC
115-165	2	22.2	7.1	165	64(147°F)	190	205	200	67.2	AC
								400	68.5	AC or AL
19-27	3			Conventional Hot Corrugated				200	72.8	LL
								400	40.4	AL
								600	Medium fractured	
412-564	3			Conventional hot corrugated				200	82.6	LL
90	3			Conventional hot corrugated				200	73.6	CC
								400	55.4	AC
								600	14.0	AA
115-165	3			Conventional hot corrugated				200	91.0	LL
								400	57.6	AL
								600	24.6	AA

LOF = Locus of failure, AA = Adhesive, AC = Adhesive-medium interface, CC = medium, AL = Adhesive-liner interface, LL = liner.

1. Addition of 0.7% borax (based on starch), and
2. incorporation of alkali to provide a pH of approximately 10.

Application of alkaline adhesives was considered a means of improving penetration into sized components, whereas borax was selected for its complexing ability with carbohydrates.

Figure 7 illustrates the results of these trials as pin adhesion values obtained with a representative medium. As can be seen, all adhesives showed some decrease in pin adhesion strength with increasing speed, but the dropoff was most dramatic with the conventional heat-set adhesive. Other trials, run with different mediums, showed similar results.

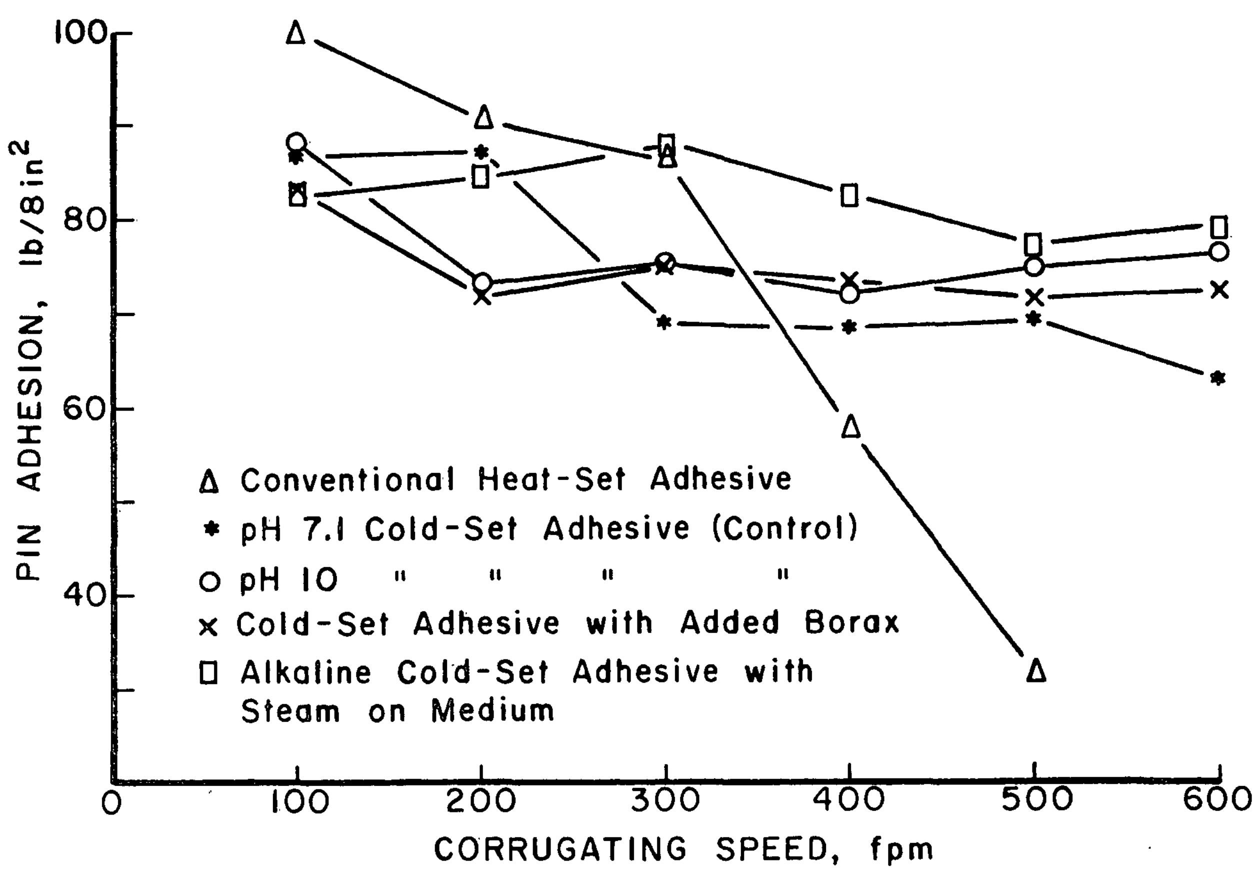


Figure 7. The effect of various adhesive modifications on pin adhesion.

c. Adhesive Brittleness

As a means of comparing the apparent brittleness of the experimental adhesives, films of hot adhesive were cast on Teflon sheeting using a coarse (40-mil) fixed clearance bar. These films were allowed to air dry at 73°F and 50% RH during which time they released from the Teflon. The apparent flexibility was judged subjectively by flexing the film until it fractured. Actually, all adhesive films prepared from the persulfate-modified starch tended to be brittle regardless of the adhesive formulation. The most flexible films were provided by the alkaline adhesives, but these were only moderately better than other formulations.

Consideration was then given to (1) incorporation of wood fiber in the cold-set adhesive, (2) addition of polyvinyl alcohol, and (3) reduced chemical modifier. All of these modifications were examined in preliminary work with small batches of adhesive.

Addition of kraft fiber was considered a potential means of reinforcing the adhesive in the bonded area. For this purpose unbleached softwood kraft pulp at 660 mL CF was incorporated directly into the adhesive slurry prior to jet cooking.

The effect of polyvinyl alcohol on adhesive properties was also tested. Elvanol 72-60 (E. I. du Pont) was dusted into the adhesive slurry in amounts ranging from 1-5% based on starch. The PVA was subsequently cooked with the starch in the jet-processing step.

The effect of reduced chemical modifier was examined over a wide range of conditions with respect to persulfate and alkali levels. Sodium sulfite was eliminated from all formulations to test the possibility that the inherent brittleness of the cold-set adhesive was due to the high level of modifiers employed in the formulations. When the adhesive became acidic through reduction or elimination of alkali, the resulting viscosities and gelation temperatures increased dramatically. However, as the cooked adhesive aged at the holding temperature of 85-90°C, the viscosity and gelation temperature declined to normal values.

The selection of adhesives was necessarily based on viscosity and gelation temperature, since these govern the practical runnability of the adhesive. Adhesives were prepared for testing with viscosities between 160-200 BU and gelation temperature between 62-65°C to ensure good runnability. Adhesive metering system clearances of 0.010 and 0.012 were used. Adhesives containing kraft fiber could not be satisfactorily applied because the fibers tended to accumulate under the metering bar thereby greatly reducing the effective adhesive film thickness. Accordingly, the amount of fiber carried with the adhesive into the bonded area was not representative of the original formulation.

The effect of these modifications is shown in Fig. 8. It is evident that none of these formula changes had a significant effect on adhesive performance.

d. Water Resistant Adhesive

A final series of corrugator trials was conducted with acidic cold-set adhesive formulations to test the possibility of forming water-resistant bonds. As a preliminary to this series, adhesives containing 0.2% persulfate were allowed to age 1-1/2 to 3 hours at 85-90°C (185-194°F) followed by the addition

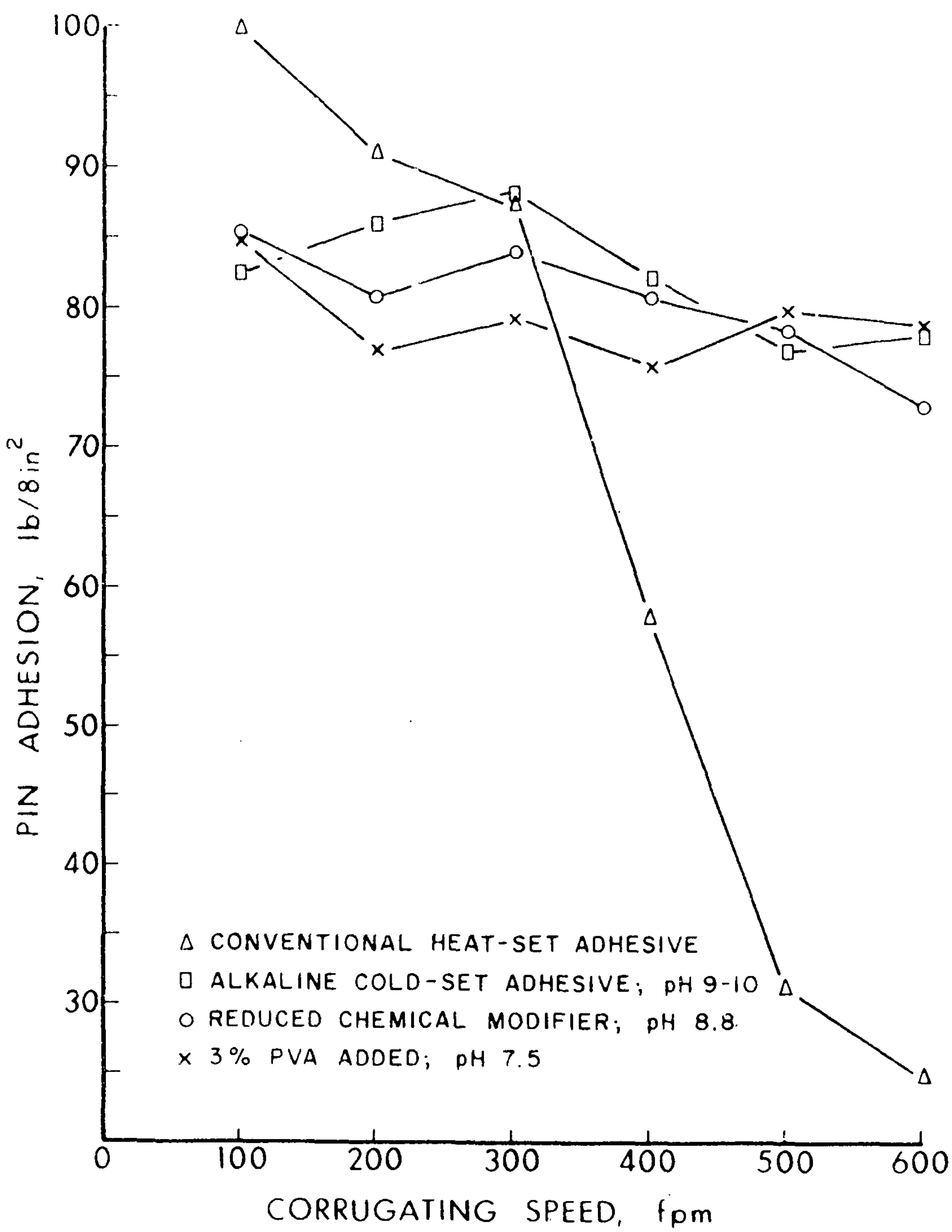


Figure 8. A comparison of cold-set adhesives (steam on medium, 0.012 inch clearance).

of low levels of cross-linking agent or resin. The aging procedure was necessary to provide a sufficiently low initial viscosity for corrugating. (Addition of cross-linking agent prior to jet cooking generally produced gelation in the jet-processing step.) Glyoxal, urea-formaldehyde (UF) and melamine-formaldehyde (MF) resin as well as polyvinyl acetate (PVAc) were added in amounts ranging from 1-10% based on starch. Only low levels of cross-linking agents could be tolerated without excessive increases in viscosity.

Corrugator trials were carried out using adhesive containing 3% of glyoxal, 1% of UF resin, and 10% of PVAc. A control adhesive prepared without cross-linking agent was included in this series. The corrugated board made from these adhesives was subsequently tested for pin adhesion after conditioning at 50 and 85% RH. It was evident in these tests that only limited water resistance was attained, probably because of (1) the inability to add 5-10% of cross-linking agent (glyoxal or UF resin) as is normally done in insolubilizing starch, and (2) the lack of heat for curing the resin. However, some water insensitivity did develop among the acidic starch formulations. In comparison with the alkaline adhesives, the acidic adhesives produced lower dry pin adhesion values but better strength levels under higher humidity conditions. This holds for adhesive with or without cross-linking agents. Adhesives containing the polyvinyl acetate resin provided roughly equivalent moist/dry adhesion ratios, but low dry pin adhesion values. The best overall results were obtained by adding glyoxal to the cold-set adhesive which improved both dry and moist adhesion.

e. Adhesive Stability

Results of the adhesive aging experiments indicate that neutral and alkaline cold-set adhesives remain reasonably stable with respect to viscosity and gelation temperature for at least 1-2 hours if the temperature is maintained at 185-190°F. Increases in viscosity related to increases in solids content become more pronounced as the initial viscosity exceeds 200 Brabender units. In contrast, strongly acidic adhesives were unstable, presumably due to continued chemical modification which occurs after the short cooking time under pressure. It is assumed that this problem would be greatly reduced or eliminated in a jet cooker designed for persulfate modification, i.e., one providing a longer dwell time.

Temperature cycling results indicate that moderate temperature fluctuations over short time intervals do not cause serious increases in viscosity. For initial viscosities in the range of 150-175 Brabender units, a temperature drop of 16°F followed by reheating resulted in a viscosity increase of only 15 Brabender units (about 10%). However, adhesives with initial viscosities exceeding 200 Brabender units are much more sensitive to temperature drops or temperature cycling.

3. Phase 3 - Technical Feasibility Studies 1975-1976

a. Introduction

This phase of the project was initiated to explore the technical feasibility of a "cold" corrugating process. Cold corrugating involves the proper treatment of the medium - e.g., with "lubricant" - so that it can be fluted satisfactorily using corrugating rolls at room temperature. This process also requires an economical adhesive which sets without heat such as the "cold-set" starch adhesive discussed earlier in this section of the report. The previous adhesive development work described above had been carried out with hot corrugating rolls. Taken together, these process elements could (a) significantly reduce energy costs, (b) reduce capital investment requirements, (c) reduce personnel heat exposure, and (d) lead to reductions in corrugator noise.

Part I of this phase was aimed at the development and optimization of cold forming techniques. Various candidate lubricating agents were screened to determine their friction characteristics under room temperature conditions. The best agents were selected for trial on the corrugator and then blended with other agents in order to improve corrugator operation. In addition, a limited study of the effects of various corrugator operational variables was carried out in order to determine the best conditions for cold forming. These results were presented in Part I of this section.

Part II of this phase was devoted to further development of the cold-set starch adhesive for cold corrugating. This required modification of the adhesive formulation developed for normal (hot) corrugating conditions because the cold corrugated medium (and liner) exhibited adhesive requirements somewhat different from those for "hot" conditions. Particular attention was given to four areas:

1. Adhesive film flexibility,
2. adhesive wet tack,
3. adhesive viscosity effects, and
4. medium receptivity effects.

b. Adhesive Film Flexibility

Materials examined in efforts to improve flexibility included glycerin, polyvinyl alcohol (PVA), borax, and PVA + borax. The combination of PVA and borax produces a complex which gels under moderately alkaline conditions. PVA

(Elvanol 72-60 - E. I. du Pont Co.) was prepared as a 4% dispersion in water by cooking over steam. Borax was added as a 2% solution. All materials were added at elevated temperature to aliquots of the precooked starch held at 185-190°F in a Dewar flask. Films of hot adhesive were cast with a 40-mil clearance bar on 1 1/2-inch wide Teflon tape mounted on 1/4-inch plate glass. The films were allowed to air dry overnight at 73°F and 50% RH and then cut into strips for flexibility measurements. Efforts to measure the degree of flex at failure of a film strip held as a cantilever beam were not successful. In general, the films tended to warp or curl as they released from the Teflon, making it difficult to obtain uniform specimens of suitable size for testing. Slight flaws in the dried film usually led to rapid failure and, as a result, the data were quite erratic. The decision was subsequently made to use MIT folding endurance as a measure of flexibility, recognizing that film strength as well as flexibility would be registered in the test results. Film strips, 3/8 x 5 inches, were utilized for this purpose.

Results of the exploratory tests are recorded in Table IV. The data indicate that 10% of glycerin or 2% of PVA improve flexibility, whereas borax embrittled the adhesive and reduced or eliminated the advantage imparted by PVA. On the basis of these results, fresh adhesives were prepared from several formulations incorporating 10% of glycerin and low percentages of PVA. The "standard" formulation was utilized at the usual solids level (22-23%) and also at somewhat higher solids in an effort to offset the dilution effect of PVA and glycerin. Adhesives were also prepared from modified formulations without sodium sulfite and with reduced amounts of ammonium persulfate. Data for these adhesives are also included in Table I. In preparing these adhesives, efforts were made to

TABLE IV
THE EFFECT OF MODIFIERS ON ADHESIVE FILM FLEXIBILITY

(Standard cold-set starch formulation)

Adhesive Components % Based on Starch					Adhesive Starch Content, %	pH	Brabender Viscosity at 95°C & 190 rpm Units	Gelation Temperature, °C	Dry Film Thickness, mils	Apparent Film Flexibility (MIT Folding Endurance) Double Folds
AP	Sodium Sulfite	NaOH	Glycerin	PVA						
0.5	0.5	0.3	--	--	22.6	7.7	160	64-65	5.4	5
0.5	0.5	0.3	10	--	24.1	7.7	140	63	5.2	9
0.5	0.5	0.3	--	2	20.7	7.7	130	61	4.5	13
0.5	0.5	0.3	--	--	23.1	7.6	175	65	5.0	4
0.5	0.5	0.3	--	--	24.3	7.6	190	67	4.9	3
0.5	0.5	0.3	--	--	25.6	7.6	270	71	5.4	3
0.5	0.5	0.3	10	--	26.3	7.5	200	67	5.5	6
0.5	0.5	0.3	--	2	22.5	7.7	180	65	5.1	5
0.5	0.5	0.3	--	5	19.4	7.6	130	59	4.6	7
0.5	0.5	0.3	--	5	20.8	7.6	185	64	5.1	5
0.42	--	0.5	--	--	22.4	8.4	175	67	5.2	13
0.42	--	0.5	10	--	23.8	8.3	190	68	5.1	12
0.42	--	0.5	--	2	20.7	8.3	160	63	4.5	12
0.3	--	0.5	--	--	20.3	9.0	200	67	3.9	22
0.3	--	0.5	--	2	19.0	8.7	210	68	3.9	22
0.1	--	0.4	--	--	14.1	8.9	240	63	3.0	36

provide a viscosity of 160-230 Brabender units at 95°C and a gelation temperature of 64-67°C, since these conditions were previously found to be most effective.

It is clear from these results that glycerin and PVA improve flexibility, and borax causes embrittlement. The most dramatic improvements were obtained, however, by eliminating the sodium sulfite and reducing the solids content. The latter effect may result from having more water available during bond set-up.

c. Adhesive Wet Tack

Adhesive wet tack was measured empirically using an inclined plane technique. A film of freshly prepared adhesive (22-23% solids) was cast with a 12-mil clearance draw down bar on plate glass which was positioned on an inclined plane having a slope of slightly less than three degrees. A steel roll 2.5 inches in length and 1 inch in diameter (wt. 255 g) was stationed six inches ahead of the film on the inclined plane. Five seconds after the film was cast, the roll was released and the distance it traveled after contacting the film was considered a measure of wet tack, i.e., the shorter the distance traveled, the higher the tack.

A number of materials were considered as potential tackifying agents for the cold-set adhesive, including polyvinyl acetate resin, modified wood rosin, rosin-oleic acid mixtures, borated dextrin, and protein. Water-insoluble resins were selected with softening points less than 185-190°F, which is the approximate temperature of the adhesive when applied to the medium. When agents of this type were used, 0.1% of an emulsifying agent (based on total weight of adhesive) was added to the hot starch prior to adding the resin.

Most of the water-insoluble resins proved unsatisfactory for the persulfate-modified starch system. In general, the viscosity of these materials at 185°F was too high for satisfactory blending. The only exceptions to this were blends of wood rosin and oleic acid which proved moderately compatible with the starch under the conditions employed.

The emulsifying agent was eliminated when adding the borated dextrin and protein. The addition of 7.5 and 15% of borated dextrin increased overall adhesive viscosity to a level far in excess of that considered acceptable for cold corrugating. Accordingly, of the materials tested, only the protein (hide glue) and the rosin-oleic acid mixtures were sufficiently compatible to be tested in films for their effects on wet tack. Neither of these produced a sufficient increase in wet tack to warrant further studies.

d. Adhesive Viscosity Effects

In pursuing optimization of the persulfate-modified starch adhesive, cold corrugating trials were carried out utilizing adhesives at 32-33% solids over a range in viscosity from roughly 200-700 Brabender units at gelation temperatures from 60-75°C (140-167°F). These variations in viscosity were produced by steam jet cooking cornstarch at 290°F with 0.3-0.8% of ammonium persulfate based on dry weight of starch. Following the cooking operation, the pH of the adhesive was adjusted to about 10 with caustic soda. These adhesives were utilized in cold corrugating at doctor roll clearances of 8 and 12 mils at speeds of 200, 400, and 600 fpm, using a rather receptive medium and a standard liner. Average pin adhesion for all corrugating speeds at a given clearance is plotted as a function of adhesive viscosity in Fig. 9.

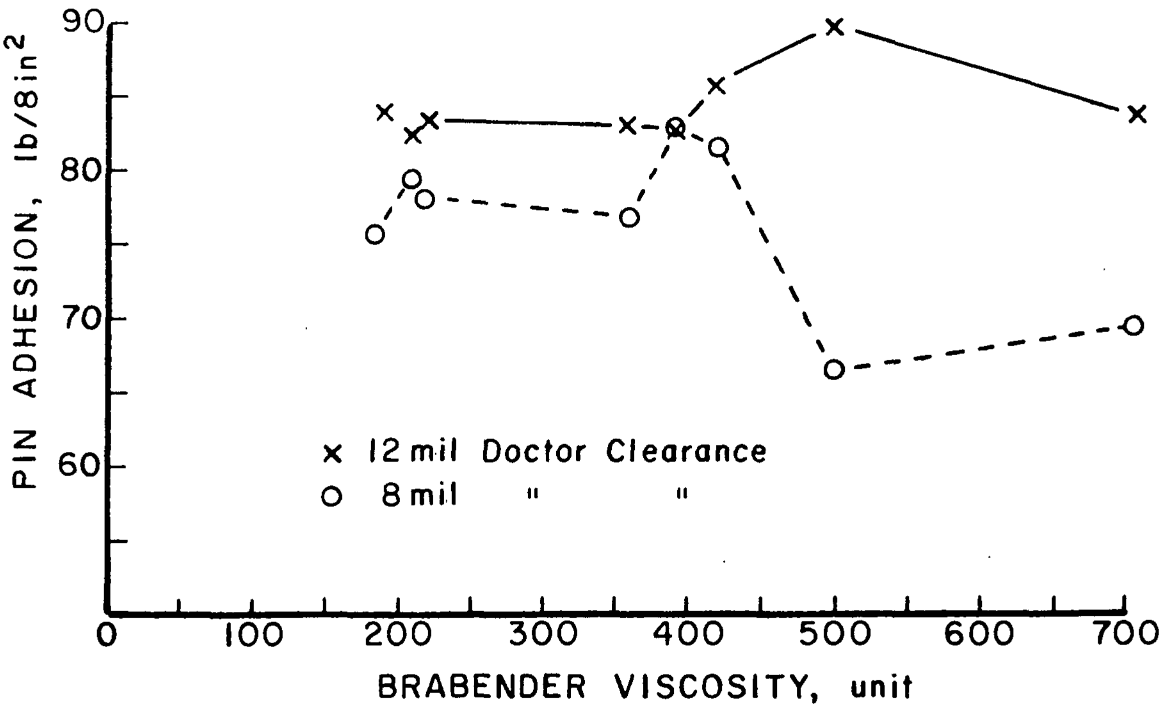


Figure 9. The effect of adhesive viscosity on pin adhesion strength (32-34% solids).

Results in Fig. 9 indicate that a rather wide range in adhesive viscosity can be tolerated at the 12-mil application level, in which case a high and rather constant level of pin adhesion, in the range of 80-90 lb, was obtained at most corrugating speeds. The predominant locus of failure in these tests was within the medium. Results at the 8-mil adhesive clearance were somewhat more varied, both with respect to pin adhesion strength and locus of failure, although some pin adhesion strengths at the 8-mil clearance were comparable to those at the 12-mil clearance. While definite relationships between pin adhesion strength and adhesive viscosity were not found, there is some indication that low or intermediate viscosity would have an advantage over very high viscosity. Presumably a high viscosity adhesive with a correspondingly high gelation temperature fails to penetrate the medium or liner adequately before gelation occurs, resulting in inferior bond quality. The reverse would be expected from very low viscosity adhesives, wherein excessive penetration would be expected to leave the surface regions deficient of adhesive. Very low gelation temperature would tend to prevent development of sufficient green bond to carry the combined board through the corrugating and cutting operations.

e. Medium Receptivity Effects

Six 26-lb corrugating medium samples exhibiting a wide range of receptivities, 58 to 1735 seconds, to liquid water in terms of the water drop test were evaluated.

The mediums were combined with a 42-lb kraft liner of commercial manufacture made within the past year.

A commercial grade of pearl cornstarch was employed in preparing both the cold set and regular starch adhesives.

The six corrugating mediums were fabricated into single-faced board using the "standard" 42-lb kraft liner at the following conditions:

1. Two adhesive viscosity levels, about 210 and 360 Brabender units
2. Two adhesive doctor roll clearances, 8- and 12-mils
3. Three fabrication speeds, 200, 400, and 600 fpm

To corrugate the mediums without fracture, under "cold" (room) temperature conditions, the mediums were treated on each side with an agent having the following composition:

Mobil Wax 130: 93%

Stearin: 5%

Dow Silicone Oil No. 200: 1%

Dixon Graphite 1110: 1%

The agent was cast into bars and abraded onto the surface of the medium.

The results of these tests are contained in Table V. Figure 10 is a plot of the grand average pin adhesion values vs. water drop. From the data obtained the following observations were made.

1. High pin adhesion strengths were obtained with the cold set starch adhesives under "cold" corrugating conditions over a wide range of medium receptivities.
2. The pin adhesion strengths obtained with the 210 and 360 unit Brabender viscosity cold set starch adhesives under "cold" corrugating conditions were not significantly different. The two adhesive viscosity levels were included in the study because of the possibility that different viscosities might be required depending on the medium receptivity. However, it appears that adhesive viscosity in the range studied is not a critical factor over a wide range of medium receptivity.
3. For the cold set starch adhesives, pin adhesion strength increased significantly as the corrugating speed decreased and the doctor roll clearance (and hence amount of starch applied) increased.

TABLE V

PIN ADHESION RESULTS USING COLD-SET STARCH ADHESIVE
ON MEDIUMS OF VARYING RECEPTIVITY

Water Drop, sec	Pin Adhesion, lb/8 sq inch: Metering Roll Clearance 0.008"				Metering Roll Clearance 0.012"				Grand Av.
	200 fpm	400 fpm	600 fpm	Av.	200 fpm	400 fpm	600 fpm	Av.	
360 Brabender Unit Adhesive Viscosity									
58	104.4	92.9	76.9	91.4	111.2	114.0	91.5	105.6	98.5
94	98.2	104.7	73.6	92.2	107.1	103.3	102.7	104.4	98.3
148	66.2	69.9	62.5	66.2	71.2	70.1	75.1	72.1	69.2
242	91.5	91.8	86.4	89.9	96.5	93.6	91.6	93.9	91.9
772	84.0	82.8	58.3	75.0	93.8	91.1	82.8	89.2	82.1
1735	96.7	95.3	81.5	91.2	114.7	106.8	103.3	108.3	99.8
Composite Av.	90.2	89.6	73.2	84.3	99.1	96.5	91.2	95.6	89.9
210 Brabender Unit Adhesive Viscosity									
58	102.2	99.3	84.7	95.4	121.0	107.8	97.2	108.7	102.0
94	98.7	86.8	82.9	89.5	109.8	98.7	102.0	103.5	96.5
148	71.4	65.8	57.1	64.8	71.3	74.6	77.8	74.6	69.7
242	91.1	84.4	73.1	82.9	101.1	95.4	87.9	94.8	88.8
772	85.0	78.6	66.4	76.7	100.6	91.5	89.8	94.0	85.3
1735	100.5	84.4	65.3	83.4	104.7	96.9	90.8	97.5	90.4
Composite Av.	91.5	83.2	71.6	82.1	101.4	94.2	90.9	95.5	88.8
Overall Av.	90.0	86.4	72.4	83.2	100.2	95.3	91.0	95.5	--

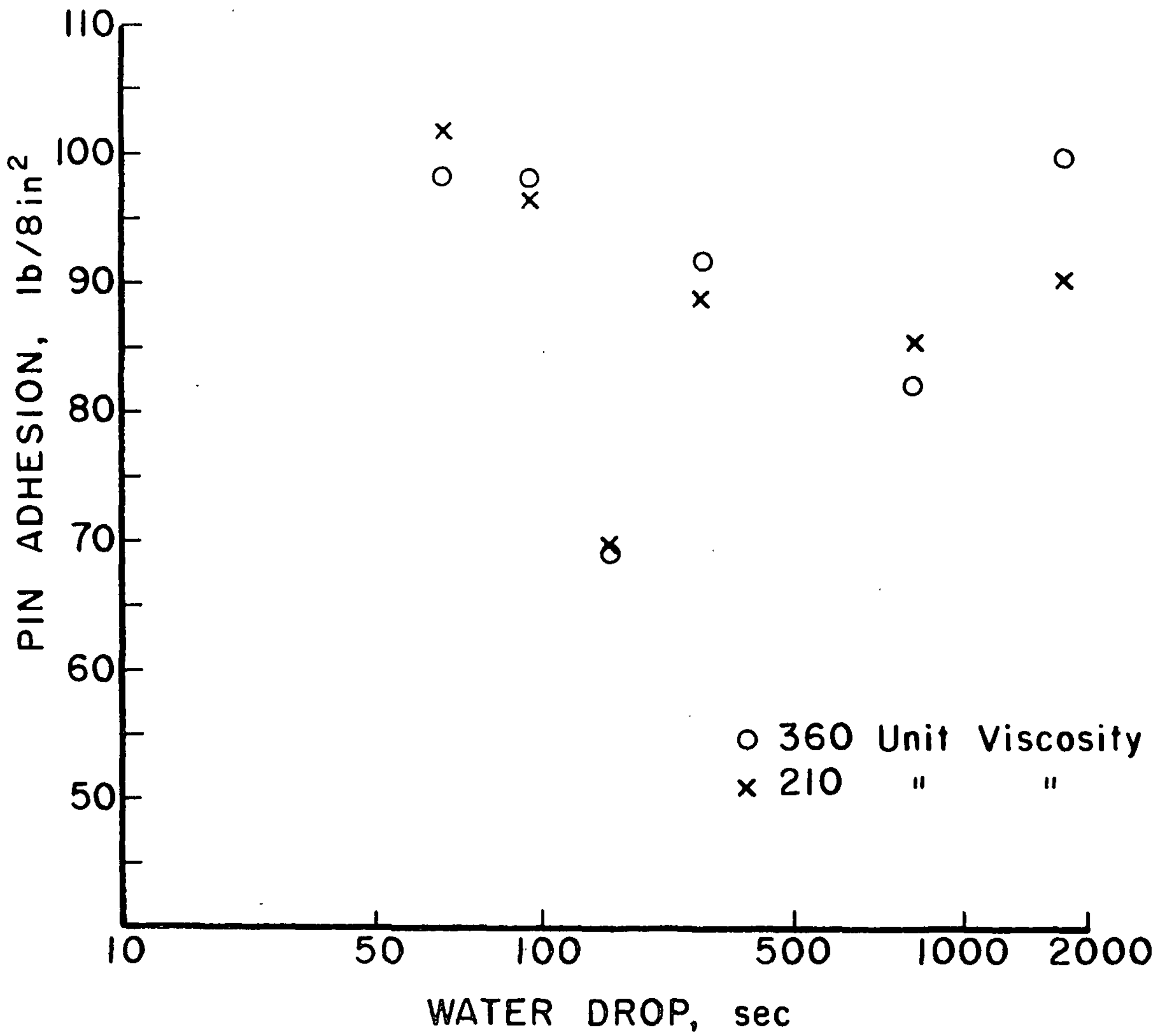


Figure 10. Effect of medium receptivity on pin adhesion strength for cold set starch adhesive.

E. BONDING SYSTEM DEVELOPMENT IN PILOT TRIALS 1977-1981

1. Introduction

Previous parts of this report have presented results from the early cold forming and bonding work. In these developments, all of the experimental work was carried out using a very crude laboratory jet cooker with a 6-second dwell time with batch addition of cooking chemicals. All bonding tests were carried out on the laboratory single facer which has a working width of 12 inches. Hence, roll stack crowning was not an issue.

Preliminary results from these trials were encouraging and demonstrated many aspects of the technical feasibility of a cold corrugating process. None of the problems of scaleup to commercial machine widths or realistic adhesive production systems was addressed, however. Futhermore, none of the development work had provided any information on the double face bonding question.

To pursue these important and unanswered questions, it was necessary to augment the laboratory with a pilot facility. At the urging of FKBG, this step was taken at a very early stage in the project, with the objective of identifying - but not necessarily solving - the real issues of commercial application of the cold process. This section of the report presents information on the important areas of bonding system development pursued during and in response to the results from the pilot trials portion of the project.

2. Laboratory and Pilot Trials Adhesive Preparation Equipment

Although numerous adhesive formulations were used in the course of the early development work, all were prepared by procedures with essentially the same elements. These procedures involved chemical additions to a pearl cornstarch slurry, jet-cooking of that slurry and, finally, adjustment of the cooked starch to a final pH by the post addition of 50% NaOH. The cooker used to prepare these adhesives lacked capacity and dwell time, and the NaOH was added in a batch mode at the conclusion of a complete cook. These features resulted in incomplete conversion, which contributed to the post cooking drift of adhesive properties, and to poor quality control. To provide suitable adhesive preparation equipment for both the laboratory and the pilot plant, two adhesive makeup systems were developed. These units, described in this section, also served as a base for defining the commercial prototype system discussed later in this report.

For the pilot trials, a jet cooker with sufficient capacity to supply the single-facer and double-backer in a commercial size machine was developed. A second similar cooker was developed for laboratory use. A schematic diagram of these cooker systems is shown in Fig. 11. Both systems were constructed from a basic jet cooker supplied by Grain Processing Corporation (GPC). Several auxiliary items of equipment were added to adapt the system to preparation of the set-back adhesive. A brief description of these systems follows.

Adhesive preparation starts by mixing a slurry of starch, water, and chemical modifiers in a large tank. A slurry mixing and holding tank, fitted with resettable flow and quantity meters to facilitate slurry preparation, was added for this purpose. Mixing of the slurry can be accomplished via a propeller-type mixer. The slurry pump can be of the Moyno or Waukesha type. The Moyno is preferred because of its smoother supply of starch to the cooking jet. Since the slurry is acidic, stainless steel is recommended for the tank, pump, and all lines.

Some measure of the slurry flow rate is required, especially for R&D purposes. Mechanical bobs or rotameters are not suitable because they are sensitive to particulate suspensions and specific gravity changes. Ultrasonic or other types of noncontact meters are also sensitive to specific gravity and hence will only be accurate at one starch solids concentration. For the pilot cooker, a magnetic flowmeter has proved satisfactory, although it requires recalibration for varying solids contents. A more suitable flowmeter is the

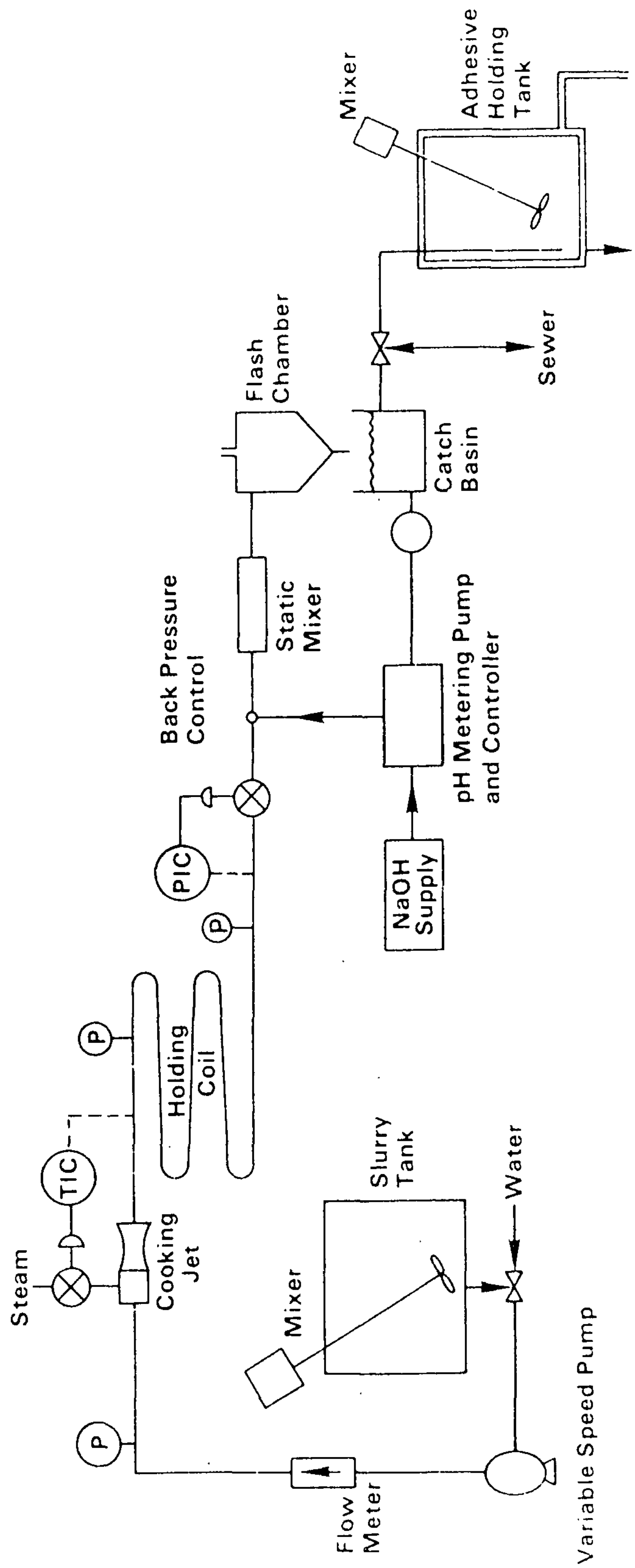


Figure 11. Schematic diagram of adhesive makeup system.

Micromotion (5) mass flowmeter. This meter, based on a coriolis concept, provides a true measure of mass flow rate, independent of the flowing fluid. It is thus unaffected by starch concentration or entrained air.

Both cookers used a specially modified Penburthy Jet, supplied by the Grain Processing Corporation. The temperature of the cooking process is determined by the amount of steam flow and the pressure in the holding coil of the cooker. In the laboratory cooker, the steam flow was regulated by a manual valve while the operator read a temperature indicator. The pilot cooker was equipped with an automatic sensor, recorder, and control valve to maintain a set temperature without operator intervention. Either system is acceptable, but the automatic system provided for more consistency and is essential for production. A cooking temperature of around 150°C is used for preparing the setback adhesive. Lower temperatures result in less starch conversion and higher adhesive viscosity and higher temperatures result in the converse.

Pressure in the cooking process was controlled by a back pressure valve at the end of the holding coil. The lab cooker used a specially modified valve obtained from the Grain Processing Corporation. Pressure was controlled by a spring in the valve which had an external adjustment for setpoint. The pilot cooker had an automatic valve, recorder, and pressure sensor. Both methods worked well and are acceptable for production use. The cooking pressure was set at 80 psi for the 150°C cooking temperature.

The holding coils for both cookers were constructed of stainless steel tubing and sized to provide a holding time of about 2-1/2 to 4 minutes. Lower holding time may prevent complete cooking of the starch and lead to a higher viscosity and possibly to a less stable adhesive. Longer holding times are allowable but are of no value in improving adhesive properties.

In both systems, a 50% solution of NaOH was injected into the adhesive stream immediately after the back pressure valve. A static mixer, located immediately downstream of the injection point, provided uniform mixing and quick chemical reaction. Injection requires a positive displacement pump to overcome the pressure drop in the static mixer. NaOH is highly corrosive and requires either stainless steel or plastic parts for the storage tank, pump, mixes and lines.

The amount injected must be accurately controlled and evenly distributed over the cooking cycle. This dictates use of a metering pump and some way of adjusting the caustic flow rate. The pump setting is determined from a measurement of the pH at the exit of the cooker. On the laboratory cooker, these measurements and caustic flow rate adjustments were carried out by the operator, as required. Once set, a good metering pump requires little attention.

The pH sensing electrode, used for pH sensing and control, was mounted in a small catch basin just under the flash chamber to minimize the time required to detect a pH change resulting from a change in the caustic rate. This eased the problem of pump control. pH electrodes for this elevated temperature environment are not dependable, thus suggesting a preference for other control approaches such as suggested above.

Except for startup and shutdown, the pilot cooker was continuous and automatic and operated with minimal attention until the need for adhesive was satisfied or the slurry supply had been exhausted. Both cookers required operator intervention for startup and shutdown. Water was passed through the cooker to preheat the equipment. When the proper temperature was reached, a manual three way valve switched the supply stream from water to slurry. The process was reversed at shutdown. The small interface volume between the water and slurry had to be discarded at both ends of a batch cook. Both systems worked well, although the laboratory cooker required more attention. Conversion characteristics of the two cookers were similar.

3. Basic Adhesive Formulation and Components

After completion and checkout, these cookers were used consistently to prepare adhesives. Because of the longer dwell time, the ammonium persulfate can react fully thus, requiring less than used in the short dwell-time cooker. Slight adjustments in cooking temperatures and pressure were also required. After these adjustments were determined and for the duration of the project, except as noted, cold corrugating adhesives were prepared as follows:

Slurry

- Pearl cornstarch - 30-42% solids
- Water - as required
- Ammonium persulfate - 0.1-0.5% on starch
- Boric acid - 0.1-0.3% on starch

Cooking

- Temperature - 250-320°F
- Pressures - 50-80 psig
- Dwell time - 2-4 minutes

Postcooking

- pH - 8-10

a. Starch

The starch used for the adhesive formulation described above is commercial grade pearl cornstarch. The moisture content of the starch can vary slightly depending on manufacturer, batch, and whether bulk or bag supplies are being used. This necessitates a check on slurry solids concentration by specific gravity or Baume' before cooking if close control of the solids content is desired. In production situations, slight variations in adhesive solids content should be permissible, thus obviating the need for trimming the slurry to a given Baume'.

A slurry solids level of 42% represents the upper limit for successful conversion in the jet cooker. This corresponds to a final adhesive solids level of about 39%.

b. Water

Limited experiments have shown that water hardness, alkalinity, and suspended solids have no appreciable affect on the normally measured characteristics of the adhesive. The pH of the water affects the final pH of the slurry. This, in turn, affects starch conversion in the cooker and alters the final adhesive viscosity and molecular weight distribution. A low pH causes excess thinning and vice versa. Consistency of the slurry pH is more important than the absolute value, although a typical range is 6.0 to 7.0. Slurry pH can be adjusted by chemical addition if it falls outside the proper range. A consistent box plant water supply should make such adjustments unnecessary in commercial practice.

c. Ammonium Persulfate (AP)

The ammonium persulfate acts as a viscosity-reducing reagent for the starch in the conversion process. The final adhesive can be described as a hydrosol mixture of amylose and amylopectin, each reduced in molecular weight from the level it had as a component of the unmodified pearl cornstarch. The amylose and amylopectin are chemically altered by the acid hydrolysis during the cooking phase (pH levels of cooked adhesive are around 2.5 to 3.0), by the oxidation due to the peroxidic structure of ammonium persulfate, and by the action of the hot alkaline environment (after pH adjustment) on unaltered glucose or already oxidized glucose units. The first two of these three chemical reactions are the direct result of the amount of ammonium persulfate added to the slurry.

The strength of the dry ammonium persulfate degrades with time when exposed to moisture in the air. Care must be taken to avoid exposing the crystals to the atmosphere for long periods of time or on a repeated basis.

The action of the AP on the starch also becomes less effective if it is present in the slurry for too long a time before cooking. In batch cooking, the AP was added with just enough time before cooking to insure complete dissolution and mixing (about 5 minutes). In continuous cooking operations, the AP was metered into the slurry stream just ahead of the cooker.

d. Boric Acid

The boric acid added to the slurry does little to the conversion process in the cooker. It helps to create an acidic atmosphere in the slurry which assists in the reduction of the size of the starch molecules and thereby lowers the amount of AP required by a small amount. Its main value, however, occurs after the cooking and post addition of NaOH where the boric acid turns into borax. The borax serves as a thickening agent and produces an adhesive with faster bond rate development.

Technical grade boric acid was used in dry form. It is much more stable than the AP and no special precautions are necessary.

e. Sodium Hydroxide (Caustic, NaOH)

As mentioned before, the addition of caustic to the hot, cooked adhesive produces further changes in the molecular size and chemical structure of the starch. The specific nature of the reactions taking place in the adhesive is unknown.

Two methods of adding the caustic have been used. In the early development work, the caustic was added to the hot adhesive in the adhesive holding tank after completion of a batch cook. This was done under atmospheric conditions and with a propeller-type mixer for agitation. The amount added was determined by measuring the pH of the adhesive in the tank. Reactions that occur after cooking and before pH adjustment change the final adhesive stability and viscosity. These variations are amplified by the poor consistency of this type of addition. In subsequent laboratory, pilot and commercial prototype systems, adhesives were prepared by injecting the caustic directly into the adhesive stream immediately downstream from the back pressure valve. After caustic injection, the adhesive flows through a static mixer, then through a flash chamber, and finally to the adhesive holding tank. Caustic addition by injection halts the acidic reactions as soon as the adhesive leaves the cooker pressure section; the static mixer section assures thorough mixing of the caustic with the adhesive. This improves long-term stability of the adhesive and batch-to-batch repeatability.

The caustic is handled as a solution of 50% NaOH in water. The details of the metering and injection system are discussed in the section on the jet cooker.

The final adhesive pH is important in terms of adhesive stability, with a pH of 9.0 to 9.4 being a typical target range.

Adhesives prepared in this fashion were characterized physically and chemically, with respect to adhesive metering for application, and for single face and double face bonding characteristics. The following sections provide details of these investigations.

4. Physical Characterization of the Cold Corrugating Adhesive

Cold corrugating adhesives are prepared by chemically modifying a pearl cornstarch slurry with ammonium persulfate. In a typical adhesive, the modified slurry is jet cooked at about 290°F for about 150 seconds and then post adjusted to pH 9 with 50% NaOH. The resulting adhesive is held at a temperature of about 190°F until it is used.

The hot setback adhesive, ready for use in the cold corrugating process, is essentially a hydrosol containing amylose and amylopectin polymer fragments. Inorganic salts resulting from the decomposition of the chemical modifier ammonium persulfate (AP) as well as sodium hydroxide and reaction products from it may also be present in the finished adhesive. In some cases, as noted, other organic and inorganic additives may be included to selectively modify the behavior and performance of the adhesive.

For setback adhesives, the development of a green bond is due in part to the change from a hydrosol to a hydrogel by cooling from contact with the cold components. There may also be a contribution from the wet tack of the hot hydrosol that is obtained even without cooling (and hence, thickening). Finally, bond strength increases as moisture is removed from the adhesive line.

Since both the bond formation processes and metering and application of setback adhesives depend critically on the physical characteristics of the adhesive material, these characteristics must be understood and controlled. The function, measurements, and importance of several key physical characteristics are described below. For convenience of reference, the list includes

1. Adhesive solids content
2. Temperature
3. pH
4. Rheology/viscosity - gel temperature
5. Penetrating ability
6. Rate of formation of bonds
7. Cohesive strength
8. Molecular weight
9. Gel temperature

a. Adhesive Solids Content

Adhesive solids content was measured by carefully driving off all of the water and measuring the remaining solid material as a mass fraction of the original quantity of adhesive. Typically, adhesive makeup slurries are set by trimming to an appropriate Baume' reading. Dilution by steam addition in the cooking process will normally drop the solids content by about 3% in the finished adhesive. Hence, for example, a 42% solids slurry will produce an adhesive at about 39% solids.

With respect to solids content several points need to be made. These are enumerated below.

a. Whatever the solids content, the final bond is stronger and the adhesive more stable with time if all of the starch present in the slurry is cooked out or thinned in the conversion process.

b. Final bond strength appears to increase slightly with solids content, although it is difficult to isolate the effect of solids alone.

c. Slurry solids levels of about 42% represent the upper limit for successful conversion in the jet cooker and this leads to a finished adhesive solids of about 39%.

d. Higher total solids levels can be achieved by using slurry fillers such as clay, although this has never proved to be effective in improving any aspect of bonding.

e. High solids levels lead to low amounts of water added during board manufacture, particularly if all the starch is dispersed to contribute to the bond. High solids levels also reduce the total amount of material to be applied and thus make adhesive metering more difficult.

f. Bond development rates increase with solids content because of the smaller amount of water that must be removed to form a bond. This has been shown with the double backer simulator, a new bond rate testing device discussed later.

b. Temperature

Setback adhesives emerge from the cooker at about 210°F and are allowed to cool to about 190°F. Cooling below this temperature results in a partially irreversible increase in adhesive viscosity and is thus to be avoided until bonding is desired. Figure 12 shows a representative viscosity-temperature cycle obtained from a Brabender Amylograph-Viscograph. From these curves, a drop in temperature of 18°C resulted in a 500 BU increase in viscosity; warming the adhesive back to the original temperature decreased the viscosity somewhat, but the net viscosity increase over the temperature cycle was 230 BU.

From a bonding point of view, it would appear desirable to hold the adhesive at a temperature near the "gel point" as defined in Fig. 12. Doing so, however, greatly increases adhesive viscosity, thus increasing the difficulty of pumping and metering. Furthermore, tests on the double backer simulator show significant evidence that lower temperatures reduce bonding. At this point, it is necessary to conclude that the adhesive temperature should be maintained near 190°F until the adhesive is applied to the flute tips.

c. pH

Adhesive pH appears to be important in terms of adhesive stability, liner wetting and penetration, and bonding. For most past work, a pH of around 9.0 has been deemed a good choice.

To provide inherent liner wetting and hence to help formation rates for double face bonds, a pH of 11.5 to 12.0 is required. Unfortunately, pH values above 11.0 are nearly impossible to reach and cannot be maintained. Thus, this approach to liner wetting is not workable with setback adhesives.

Bond formation rates and final bond strength do not appear to be strong functions of pH over the range from 7.0-10.0. It has been discovered, however, that long-term stability of the stored adhesive is best if the pH is in the

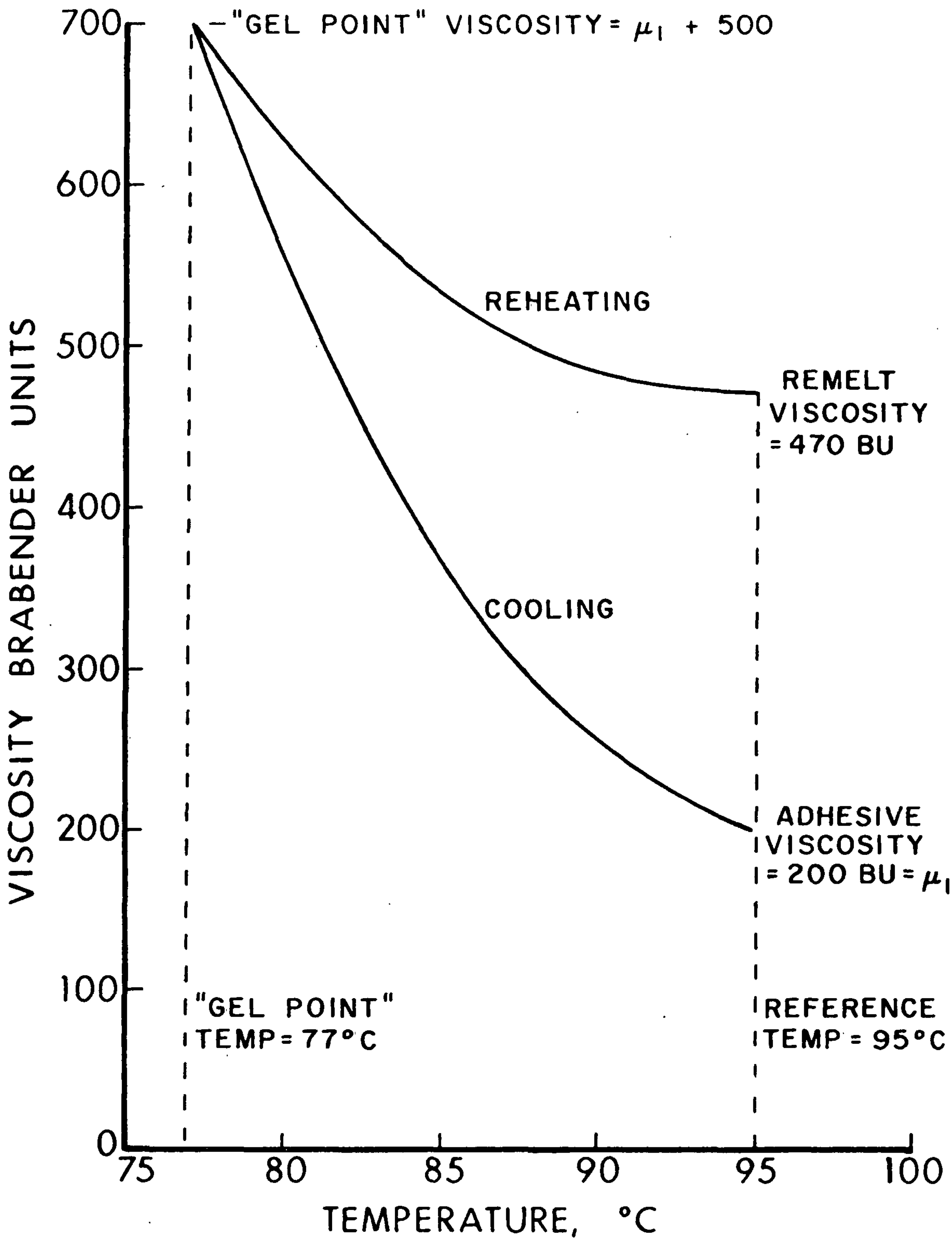


Figure 12. Adhesive temperature-viscosity characteristics.

range from 7.5 to 9.4, so this has been selected as the target range. This has obvious implications for materials of construction for adhesive system components.

d. Rheology/viscosity

The Brabender Amylograph-Viscograph has been used extensively to evaluate the low shear rate viscosity and temperature-viscosity history of setback adhesives. Representative curves and definitions are given in Fig. 12. Viscosity, as measured in this way, is an important indicator of the degree of thinning of the starch polymer and thus is an indirect indicator of the effect of thinning agents and conversion conditions. In this way it serves as a useful tool in evolving adhesive formulations with desired properties.

Brabender viscosity is also important as an indicator of the ease with which a given material may be pumped and handled in an adhesive system. For adhesive systems presently in use, best handling performance is realized if the Brabender viscosity is less than 400 BU.

At higher shear rates, setback adhesives are shear thinning. Viscosity value at high shear rate is an important property of the adhesive in determining its performance in an adhesive applicator system. A Haake RV-2 high shear viscometer, shown in Fig. 13 fitted with a special sample holding unit to maintain the adhesive under controlled temperatures, was used for the analysis of adhesives under condition of high shear rate.

Setback adhesives show a 25-35% decrease in viscosity over the shear rate range from 0 to 20,000 sec^{-1}. As indicated by the changing slope of a typical flow curve, shown in Fig. 14. By comparison, a conventional corrugating adhesive would show an 80% decrease in viscosity (slope of the flow curve) over the same shear rate range. Thus, at the high-shear rate conditions encountered in the nip of two roll applicator systems, setback adhesives have at least twice the viscosity of conventional materials. This has an important impact on adhesive metering as discussed later.

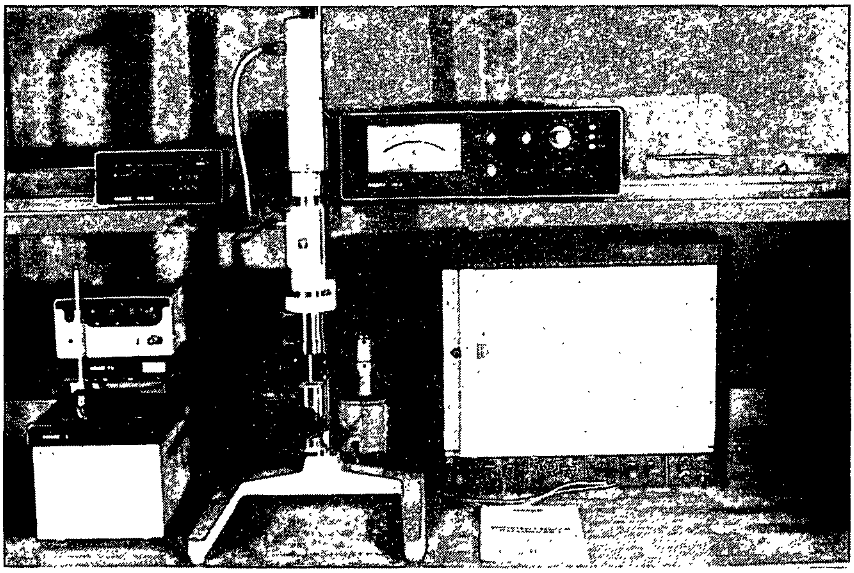


Figure 13. Haake RV-2 high shear viscometer.

e. Penetrating Ability

Penetrating ability is a measure of the depth to which starch from the setback adhesive migrates into the bonded components. It is usually measured by taper grinding or step grinding components after bonding is complete, followed by iodine staining to determine the depth to which starch is resident. Good final bond strength is believed to require penetration of at least 0.002 inch; deeper penetration may actually be wasteful in terms of adhesive consumption. Fast bond development requires that the penetration occur rapidly to quickly disperse the water in the adhesive and also to quickly cool the adhesive. No technique for measuring penetration rate is presently known.

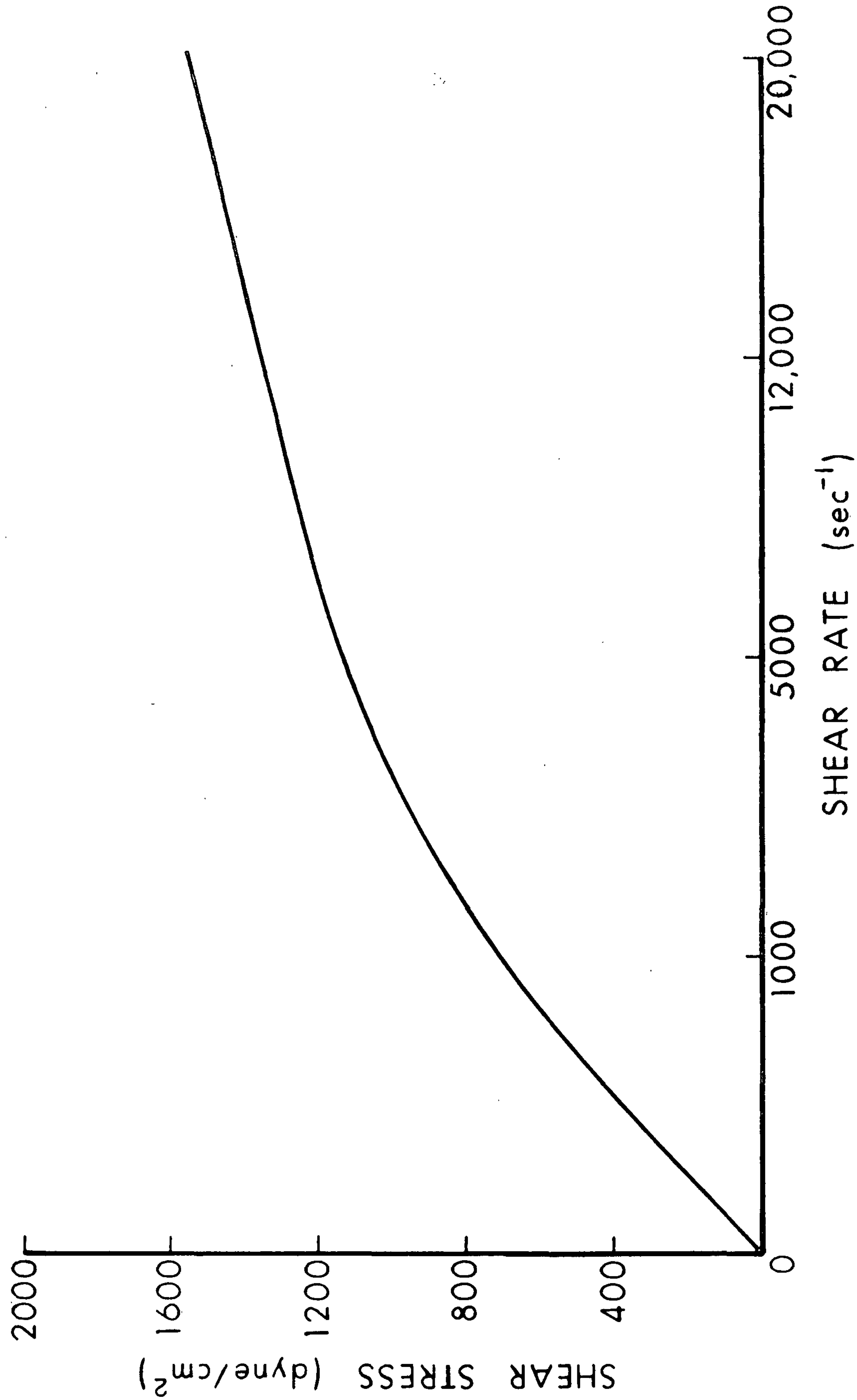


Figure 14. Haake output.

At the single facer, considerable pressure is applied during the combining operation. This results in significant mechanically induced penetration of the components with 0.004 inch being common and 0.008 inch occurring occasionally. From a final bond standpoint this penetration is not necessary. However, to develop a sufficient green bond in the short time available, it is necessary to use pressure roll loading values that result in deep penetration. It should be noted, however, that despite the deep penetration, most of the starch remains in the first 0.002 inch of the components.

At the double backer, combining pressures are too low to mechanically induce penetration. Hence only the naturally occurring gradients are available to move the starch. Typically, penetration is about 0.002 inch but nothing is known of the penetration rate. Because of the low pressure combining condition, the double facing bond formation rate may be slower than that on the single face side. This implies the need to select the adhesive and the combining conditions to optimize bond development rates.

f. Bond Formation Rates

Satisfactory production of combined board at high speeds requires an adhesive that forms a strong fiber-tearing bond in a short time so the board will withstand the rigors of slitting, scoring, and cutoff. At a production speed of 600 fpm with present machine lengths, only about 8-10 seconds are available for the bond to form. The objective then, in adhesive development, is to produce a formulation that will set, under double backing conditions, to a strong fiber tearing bond in 8 seconds or less.

Early development work was based on use of the pilot facility or a simple and subjective bench test to evaluate bond development rate. In the latter test, a thin film of adhesive was drawn down on a temperature controlled plate, a single face sample was contacted with the plate to apply adhesive to the flute tips, and then the sample was combined with a double face liner. After 8 seconds, the bond was separated and the bond site inspected for fiber tear. Neither pilot trials nor the bench test provided enough control over the experiment or precision in the bond evaluation to permit effective comparison and optimization of adhesives. Because of this, a sophisticated double backer simulator was developed and used in development of the double face bonding system. Briefly, this device permits formation of bonds under carefully controlled conditions that duplicate double backing. It also permits evaluation of bond strength as a function of bond age and thus serves as an effective tool in bonding studies. A more complete description of this instrument is given in a later section.

g. Cohesive Strength of the Adhesive Polymer

In order that adhesive joint failure during testing or commercial industrial use not take place within the film of adhesive joining the adherends, the substance comprising the adhesive must have a minimum cohesive strength exceeding the strengths of the adhesive/adhered interface or the strength of the adherend substrates.

In the early work with cold corrugating adhesives containing only AP-modified starches, the adhesive failure locus was frequently within the adhesive layer, particularly at low solids. As improvements in preparation and formulation took place (even at low solids) the locus of failure changed to adherend/adhesive interface or within the medium. With adhesives prepared in the extended dwell time cookers, the locus of failure is rarely at an interface, but more usually within the medium, and even fairly frequently within the liner.

h. Molecular Weight of the Adhesive Polymer

A wide variety of techniques and procedures is available for determining the molecular weight of the starchy adhesive polymer. The method used in this work is to calculate the polymer molecular weight by using the intrinsic viscosity fitted into the following equation:

$$[\eta] = K \cdot M^a$$

where $[\eta]$ is the intrinsic viscosity, M is the molecular weight, and K and a are parameters whose values are taken from other work and methods for determining starch (or amylose or amylopectin) molecular weights.

The intrinsic viscosity $[\eta]$ is defined by the relation

$$[\eta] = \lim_{c \to 0} (\eta_{sp}/c) = \lim_{c \to 0} (\eta_k - 1/c) = \lim_{c \to 0} [(\eta/\eta_0 - 1]/c$$

where η_k is the relative viscosity

η_{sp} is the specific viscosity

η_0 and η the viscosities of the pure solvent and polymer solution, respectively, at concentration c. $[\eta]$ usually has units of cubic centimeters per gram.

Wolff, Gundrum, and Rist developed a simplified procedure by which the relative viscosities (η_k) are measured by the time of flow of a 0.2% solution of adhesive solids dissolved in 1N KOH in a calibrated No. 100 Ostwald-Cannon-Fenske viscometer maintained at 25.10 ± 0.03°C in a thermostated bath. Present adhesive polymers require a No. 50 viscometer to give long enough flow times to yield adequate precision in the determinations. Using the equation

$$M = \frac{162\eta}{1.67 \times 10^{-3}}$$

the weight average molecular weights for several setback adhesive polymers have been found to range from 30,000 to 70,000.

A list of molecular weights of several polymers best suited for good adhesion action is shown below: [Reference (6), J. P. Casey].

Product	Mol. Wt. Range
Polyvinyl acetate	1,720-17,200
Polyethylacrylate	8,000-15,000
Polyisobutylene	2,800-8,400
Polyamides	12,900-25,800
Cellulose nitrate	51,750-103,500

Thus, the setback cold corrugating adhesive seems to be of a suitable order of molecular size.

i. Gel Temperature

This is an arbitrarily chosen term defined as the temperature at which (upon cooling under the 1.5°C per minute rate of decrease of temperature in the Brabender Amylograph-Viscograph from 95°C at a stirring rate of 190 rpm) the Brabender unit viscosity has increased by 500 units (875 cps), Fig. 12. Obviously the higher the gel temperature the more rapid is the cooling induced thickening or setting of the adhesive.

j. Adhesive Stability

Adhesive viscosity stability with temperature and time are important in determining pot life and the degree of control required in handling equipment. Table VI includes viscosity values for fresh and stored (2 hours) adhesives cooked at 140 and 150°C. Table VI also shows the effect of slight cooling following by reheating on adhesive viscosity. Ideally, viscosity should increase with cooling and then fully recover upon reheating (at least for limited cooling) so that small temperature drops in the adhesive handling system would be acceptable. The adhesive prepared at 150°C shows better adhesive stability with both temperature and time than the one prepared at 140°C.

TABLE VI

VISCOSITY STABILITY WITH TIME AND TEMPERATURE

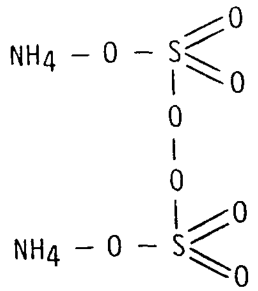
	Viscosity					
Cooking Temperature, °C	Fresh 95°C	After 2 Hours Storage 95°C	% Change	After Cooling to 75°C	After Reheating to 95°C	% Permanent Increase In Viscosity
140	330			770	510	55
150	215			540	250	16
140	385	425	10			
150	415	430	4			

5. Chemical Characterization of the Cold Corrugating Adhesive

Chemical literature references on the effect of ammonium persulfate as a viscosity-reducing reagent for starch hydrosols, combined with other references on the effects of a hot alkaline environment on starches, lead to the expectation that several chemical changes occur in the starch polymer structure and configuration. These can be best discerned by a chemical characterization of the corrugating adhesive.

a. Conversion Chemistry

The cold corrugating adhesive is a hydrosol of a mixture of amylose and amylopectin, each reduced in molecular weight from the level it had as a component of unmodified corn pearl starch. These components are chemically altered by acid hydrolysis during the cooking phase (pH levels of cooked paste are around 2.5 to 3.0), oxidative alteration due to the peroxidic structure of ammonium persulfate (AP), structurally depicted as



and the combined action of the hot alkaline environment on unaltered glucose units and/or already oxidized glucose units.

b. Carboxyl Groups

Spot-test reactions with methylene blue dye on dried particles of the cold corrugating adhesive show little or no carboxyl (-COOH) groups to be present. This observation is in line with literature references which state that persulfate oxidations of carbohydrate produce little or no carboxyl groupings (1).

c. Reducing Value/Aldehyde Groups

Aldehyde (-CHO) groups are produced at each site where the starch polymer chain is cleaved by acid hydrolysis. Since aldehyde groups are reducing agents, one can get a measure of the amount of polymer cleavage by measuring the reducing value of the adhesive. A sample of adhesive cooked at 140°C (284°F) with 0.3% AP, with a 90-second holding time, was analyzed for reducing value and found to have a value equivalent to 4.4% glucose by the Taylor, Fletcher, and Adams methodology for determination of initial reducing value (7).

The above reducing value is at first glance surprisingly low in view of the fact that the starch has been in an acidic environment (pH about 2.5-3.0) at a temperature of 140°C (284°F) for 90 seconds. However, in addition to causing polymer cleavage, the acid environment can catalyze reactions of the aldehyde reducing groups with other portions of the adhesive so that the final amount of aldehyde grouping is lowered. One such reaction is the formation of acetals, R_1-$CH(OR_2)_2$, which are resistant to hydrolysis in the final alkaline environment, thus lowering the final aldehyde content.

d. Post Cooking Conversion

In addition to the changes in molecular size and chemical structure which take place during the acidic 90-second cooking phase of the adhesive preparation, there are further changes induced by the final hot alkaline environment. It has previously been noticed that the pH drops during extended holding at 190°F (8), which indicates that acidic products are being formed by the action of the hot alkali on the adhesive.

The specific nature of the reactions taking place in the hot alkaline corrugating adhesive is unknown. There are a great number of references in the literature covering alkaline degradation of carbohydrates such as cellulose and starch (9,10-20), but they are of doubtful value in devising improvements in the cold corrugating adhesive.

e. Iodine Affinity

The iodine affinity of starch is a measurement of the binding of iodine by a solution of starch into a complex with the amylose component of starch (21). By conducting a potentiometric titration of a starch solution with 0.001*N* iodine solution, one can determine the iodine affinity which, for the amylose component of unmodified cornstarch, is 189 mg/gram. For unmodified cornstarch the amylose content i0 27%, so that one would expect to have an iodine affinity equal to 189 x 0.27 or 51.03. The iodine affinity of a sample of cold corrugating adhesive was 32.0, which represents a reduction of 37.2% in iodine binding capacity. This reduction in the ability of the amylose portion present in the starting cornstarch to assume the helical configuration required to bind iodine must be caused by changes in the chemical structure of the starch during the AP oxidation and consequent hot alkaline environment holding. The exact nature of the changes is not known for this particular circumstance.

6. Application Characteristics of the Cold Corrugating Adhesive

Conventional corrugators usually utilize a two roll adhesive applicator for both the single facers and the glue machine. At the single facer, two co-rotating smooth rolls are typical; at the glue machine, a smooth doctor roll and a gravure transfer roll are normally used. Since these systems are so common in existing machines, the desirability of their continued use with setback adhesives for the cold process is evident. The objective, of course, is to use these systems within their normal operating range and to achieve satisfactory rates, uniformity, and controllability of adhesive application. As noted earlier, setback adhesives have rheological characteristics which differ from those of conventional adhesives in a way that is significant to metering. In this section, the relationships between rheological characteristics of setback adhesives and the metering process will be explored and the inherent difficulties of metering highly concentrated adhesives with two roll systems will be pointed out. Actual operating experience with conventional applicator systems and setback adhesives will be described and, finally, the implications of the properties of setback adhesives in terms of applicator system design will be outlined.

a. Metering Processes

Adhesive application is a two-stage process in which a thin film of adhesive is metered onto a transfer roll which, in turn, comes in contact with the medium flute tips to transfer adhesive to them. If the assumption is made — and it seems to be supported by experience — that the amount of adhesive applied is directly related to the amount on the transfer roll, then effective control of application rate can be achieved by effective control of metering. With either of the two systems in dominant use, metering is accomplished by the shearing action in the nip between the two corotating rolls (the surfaces move in opposite directions in the nip thus creating the required shearing action).

In a study of the subject metering process (albeit for smooth rolls only) Jurewicz (22) established the essentials of the relationship between the metered amount and the system parameters. Film thickness was found to vary directly with metering gap and in somewhat more complicated ways with surface speed ratios of the two rolls (M/A), operating speed, and the viscosity of the metered fluid under high shear rates. It was further discovered that for a fluid with a given high-shear viscosity, there is a roll surface speed ratio at which film thickness is independent of operating speed. This, in turn, implies an adhesive application rate that is independent of machine speed and that can be readily and directly controlled by gap adjustments.

Based on these findings, Jurewicz recommended that the high-shear viscosity of corrugating adhesives be monitored continuously, that the speed ratio be set to the corresponding critical value, and that application be controlled by gap setting. For conventional adhesives, the high-shear viscosity is around 15 cp, the critical M/A is about 0.9, and gaps of the order of 6-10 mils are required.

Setback adhesives have rheological properties that are qualitatively similar to conventional adhesives (shear-thinning) but, quantitatively, the setback adhesives have much higher high-shear viscosities. This has a significant effect on metering as shown by the data in Fig. 15. These data show that the critical speed ratio is low (< 0.4) and that the film thickness is approximately equal to the metering gap. This, coupled with solids contents of 32-39%, suggests that metering gaps of the order of 3-5 mils may be required. This will be very difficult to achieve in commerical machinery, thus suggesting the serious consideration of alternative metering and application systems. The dynamics of metering against gravure rolls have not been studied, but these and other problems are believed to be present there as well.

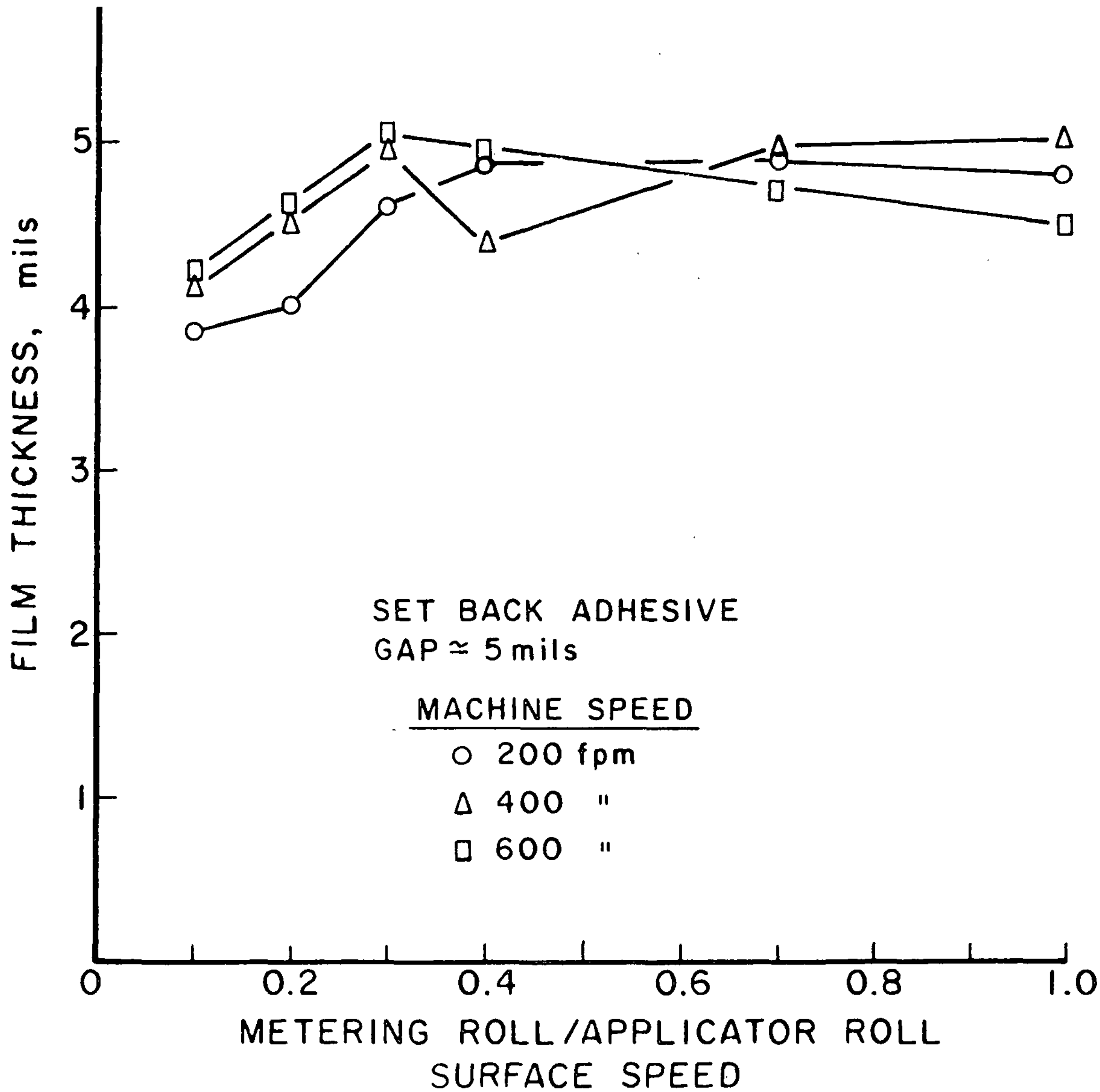


Figure 15. Metering system characteristics for setback adhesives.

b. Transfer Processes

The nature of the tranfer roll, smooth or gravure, determines the distribution of adhesive on the flute tips. This is shown in Fig. 16. Contact with a smooth roll tends to squeeze the adhesive off the tip and onto the shoulders of the flute. The gravure roll transfers adhesive from cells to the flute tip where it remains to provide a uniform coverage. More adhesive will be applied if a film is allowed to remain on the surface of the gravure roll. At the single facer, bonding is dependent on moisture transfer induced by the high pressures generated as the pressure roll is forced against the lower corrugating roll. The absence of adhesive at the center of the flute tip resulting from the use of a smooth applicator roll may impede this process and reduce the efficiency of bonding. If so, a gravure roll would be better at the single facer. However, as later results will show, the redistribution of the adhesive at the pressure roll nip may be more important than the initial distribution from the glue roll. A gravure roll is used at the glue machine, since bonding rate and bonding efficiency (bond strength per unit of adhesive) are both maximized by applying a thin uniform film to the flute.

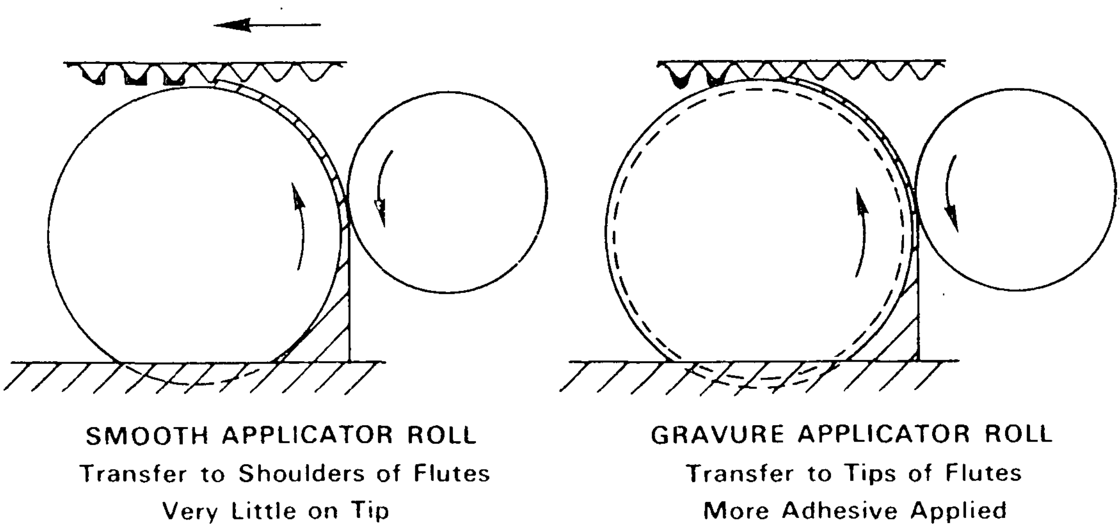


Figure 16. Application of adhesive to flute tips.

Excess adhesive affects hot and cold corrugating in a similar manner. Increased consumption means increased cost. More adhesive means more added water which leads to increased warp and washboarding. The extra water also slows double face bonding. In the hot process, compensation can be made by increasing the heat or the amount of contact of the board with the steam chests or reducing speed. In the cold process, reduced running speed is the only compensation available. Finally, extra adhesive usually yields only modest increases in bond strength, once a certain minimum has been reached. These factors make it especially important to have good control over the metering and application processes in cold corrugating.

c. Operating Experience

The IPC pilot single facer has a two roll applicator system with speed ratios (M/A) variable down to 0.35. On the pilot corrugator, the two roll applicator system is geometrically similar but the speed ratio is fixed at 0.8. Both operate with gaps as low as 5 mils, but this is probably below the practical limit for commercial operation of a wide machine. Minimum achievable application rates for the 12-inch machine (M/A = 0.35) range around 1.0-1.2 pounds/M ft^2, whereas those for the wide machine (M/A = 0.8) are always above 1.5 pounds/M ft^2. Thus, both rates are too high, but the higher M/A definitely contributes to thicker films and more adhesive on the flute tips.

At the glue machine, metering is normally accomplished by a smooth, chrome-plated doctor roll working with a gravure roll with 16 cells/inch. Each cell is 0.018-inch deep. For setback adhesives, this system gives completely unsatisfactory metering. At gaps above about 6 mils, the transfer roll carries a thick layer of adhesive and transfers far too much to the flute tips. At gaps below about 5 mils, the gravure roll is stripped of all transferable adhesive so none is transferred to the flute tips. It thus appears that a smooth roll-gravure roll system is not workable with setback adhesives.

These issues will be explored much more completely in the later sections on pilot and commercial prototype trials.

7. Adhesive Bonding Characteristics

In previous sections of this report the physical, chemical, and application characteristics of setback adhesives have been described. None of these characteristics has any relevance, however, unless the bonding properties of the adhesives are acceptable. Bonding strength must be viewed in terms of the time rate of bond development, the ultimate strength of the bond, the locus of bond failure, and the stability of these characteristics with age and environment. These characteristics must also be sufficiently insensitive to component properties to permit successful bonding for the normal range of roll stock variations. The purpose of this section is to present the bonding properties of various setback adhesives and to point out areas where further improvements are needed.

a. Final Bond Strength

Final bond strength is a measure of the force required to disrupt the bond joining the medium and liner. For a good adhesive, this strength must be at least 10 psi (3 lb/inch of flute) and the bond must fail by pulling fiber from the medium or the liner. If the bond fails by pulling fiber, then the cohesive strength of the adhesive is greater than the strength of one or more of the components and the adhesive has penetrated sufficiently to provide maximum bond strength. Both are important properties. Failure in the adhesive, i.e., without pulling fiber, implies that the adhesive is the weakest link in the bonding system. Final bond strength is typically evaluated by a standard pin adhesion test or by testing mature bonds on the double backer simulator discussed later.

Many different setback adhesive formulas have been evaluated, with most giving excellent final bond strength, usually at least 5 lb/lineal inch. A few have been on the brittle side with a consequent adhesive failure rather than a fiber failure. Bond strengths are usually nearly the same for single face and double face bonds. At bond failure, fiber is almost always pulled from one component or the other, with medium fiber failure being the more common. Suffice it to say that many setback adhesive formulas can be devised that give strong, fiber pulling final bonds for either single facing or double facing use. Required application rates will be discussed below.

b. Bond Development Rates

Adhesives suitable for corrugating must develop nearly full bond strength in a very short period of time so the freshly manufactured board has sufficient strength to withstand subsequent processing operations, particularly slitting, scoring, and stacking. In hot corrugating, bond development results from heat-induced gelatinization and drying of the adhesive, a process that occurs under large heat transfer gradients imposed externally by the hot plate section. Setback adhesives develop bond strength through loss of heat and moisture, but in the cold process, particularly in the double backer, these transport processes cannot be forced externally and thus must occur under natural gradients. To achieve satisfactory bond development rates under these conditions, it is necessary to minimize the amount of cooling and drying required to form a bond while simultaneously maximizing the transport rates.

Machinery set up for the normal production of corrugated board is poorly suited to the evaluation of bond development rates. In the pilot facility described later, for example, it was possible to observe whether the bonds were sufficiently strong to withstand slitting and scoring under given operating conditions. It was not possible, however, to determine quantitatively the strength of the green bond nor was it possible to determine anything about the bond before or after the slitting point. In short, such facilities are expensive to use, work under poorly controlled conditions, and yield little useful information for absolute or comparative evaluation of bonding system performance. These factors, coupled with the difficulty of achieving satisfactory bonding rates under the imposed conditions, led to the development of a double backer simulator as an adhesive development tool. A description of the simulator is given below, followed by more specific information on the bond development rates of setback adhesives.

In the early parts of this section, the general characteristics of a basic setback adhesive for cold corrugating were given. This formulation, or one nearly like it, was used in the early pilot trials to identify the critical issues for commercial operation of the cold process. It was also the basic starting point for laboratory exploration of numerous variants on the adhesive formulation.

For the early pilot trials two tentative conclusions were drawn about the setback adhesive:

1. Single face bond performance appeared adequate for commerical operation, although the true performance of the adhesive was somewhat masked by the excessive

adhesive application rate characteristics of the pilot single facer. For this reason, little additional attention was paid to SF bonding until the Savannah trials were well along. In those trials, better application equipment allowed lower application rates which finally revealed the brittle nature of the single face bond. Investigation of this problem is discussed later in this section.

2. All of the double facer bonding results showed clearly that faster DF bonding would be necessary to satisfy commercial production requirements. Considerable laboratory work, discussed in the immediately following parts of this report, was devoted to this issue.

Based on observations in the pilot trials and the general economic requirements for the cold corrugating process, the following basic bonding specifications were set.

a. Required application rates should not exceed about one pound of starch per 1000 sq feet of liner or two pounds per 1000 sq ft of single-wall combined board.
b. Adhesive cost should not significantly exceed the cost of dry pearl starch.
c. Bond development rates should be sufficient to result in a firm, fiber-tearing bond within 8 seconds so the board will withstand the sheeting operations and be reasonably stiff for convenient handling. For normal

double backers operating at about 600 fpm the elapsed time through the machine is about 8 seconds, hence the bonding time requirement above.

d. Adhesive rheology must be compatible with conventional applicator systems or minor modifications thereto.

e. Adhesive preparation, handling, and cleanup must be consistent with normal box plant operations.

f. At the application rates specified in (a), final bond strengths should approach 10 psi (3 lb/inch of flute).

Of these requirements, bond rate enhancement appeared to be the most significant hurdle to achieving commercially realistic operation of the cold process at the time the pilot trials were conducted.

Setback adhesives develop bond strength partly by cooling and partly by drying. It is apparent that, for double-face bonds, the contribution of cooling develops quickly, but does not yield sufficient strength to withstand the rigors of high-speed slitting and scoring. It is equally apparent that moisture removal occurs slowly under typical operating conditions, but is the dominant factor in developing a green bond, with present or similar adhesives. One thus concludes that faster bonding requires increasing the moisture transfer rate across the adhesive-liner and/or adhesive-medium interfaces. Of these, the liner interface seems more important. Alternatively, the adhesive must be modified to reduce the amount of moisture loss required to form a bond. Both approaches provided avenues for adhesive development work.

8. Adhesive Development

At this point in the project (early pilot trials), the search for a cold corrugating adhesive which would quickly develop an adequate green bond and produce a fiber failure in the liner surface was carried out by means of crude, subjective test procedures. A film of hot adhesive was drawn down on a brass plate positioned over a tray of boiling water (to maintain a surface temperature of 190-195°F) with a Gardner bar adjusted to produce a 0.010-inch wet film. A section of single-face board, approximately 2 inches wide and 4 inches long, was placed onto the wet adhesive film to apply glue to the flute tips. The single face sample was then placed in contact with the felt side of a piece of linerboard, held in place by double-sided tape. Weights on the single face sample were used to control combining pressure.

The adhesive joint could be formed immediately or after any desired open time. After forming, the joint could be allowed to mature, undisturbed, or pulled apart manually after any length of contact to observe the degree of bonding and/or type of failure of the bond. Quantitative measures of the bond breaking force could not be obtained.

In this series of experiments the search for improved formulations included the following approaches:

1. Adding boric acid to the slurry to form borax in the final, hot, alkaline adhesive.
2. Increased starch slurry solids contents (for higher adhesive solids content).
3. Alkali preswelling of starch in the slurry at normal solids levels.
4. Alkali preswelling of starch in slurries with higher solids levels obtained through addition of clay.
5. Adding polyvinyl alcohol to the slurry.

6. Combined addition of polyvinyl alcohol and clay to raise the slurry solids level.

For each of these experiments, the slurry was pumped to the jet cooker at 3.25 gpm, cooked for 90 seconds at 140°C (284°F), flashed to atmospheric pressure and discharged to a holding tank at 195-200°F. Sufficient ammonium persulfate was included in the slurry to get the desired final adhesive viscosity. Sodium hydroxide (50%) was injected into the adhesive stream in a static mixer at the exit of the cooker. The final pH was set in the range 8.8-9.2.

Described below for each different formulation are the rationale behind the approach and the expected results, the specifics of the formulation, and the properties and performance of the resulting adhesive. The crudeness of the bond test procedures must be kept in mind in assessing the results. However, at the time these experiments were conducted, a doubling of adhesive bond rate was desired. A change of this magnitude would have been revealed, even by the crude test.

a. Boric Acid Addition (Table VII)

TABLE VII

SLURRY COMPOSITION

Water	78 liters	(33% Starch solids)
Boric acid (technical grade)	82 grams	(0.25)
Starch	100 pounds	(about 10% H_2O)
Ammonium persulfate	123 grams	(0.3%)

The Brabender viscosity of the resulting adhesive was in the range from 250-280 units and remained relatively steady on holding for several hours. The "gel"* temperature was about 80°C (176°F).

The addition of caustic soda to produce a pH of 8.8-9.2 converted the boric acid to borax. The borax is believed to provide increased flexibility and toughness of the cold adhesive film, with probable improvements in pin adhesion values in pilot single-facer runs. Quantitative improvements in bonding performance were not discernible with the test methods available at the time, although the borated adhesives seemed to remain more fluid and, thus, easier to handle and clean up. Cost is not a significant factor because of the low addition rate. Boric acid is a stable, easy to handle chemical. It was included in almost all formulations used subsequent to these early tests.

b. Increased Solids (Starch) Levels (Table VIII)

Increasing the solids levels in the adhesive was expected to increase the bond development rate and, at the same time, reduce the amount of water added to the board. Only small increases in solids were possible, however, before dilatancy and uncooked starch granules were encountered. These early experiments were conducted at slurry solids levels of about 41% as compared to 33-36% for most of the prior work.

Because of the crude bond evaluation procedures in use at the time, no well defined benefit over the lower solids adhesives could be found. Even borated high solids adhesives were no better than borated adhesives at the normal solids levels. These results, coupled with anticipated difficulty in handling the

*Gel temperature is defined as the temperature to which the adhesive must be cooled to produce a 500 BU increase in viscosity from a starting point of 95°C. All measurements were made at a spindle speed of 195 rpm.

higher solids adhesives, delayed their use until later when more quantitative simulator data conclusively proved their value.

TABLE VIII

SLURRY COMPOSITION

Water	55 liters	(41% solids)
Boric acid	82 grams	(0.2%)
Starch	100 pounds	(about 20% H_2O)
Ammonium persulfate	164 grams	(0.4%)

c. Alkali Preswelling of Starch in Slurry (Table IX)

In some cases the presence of some uncooked raw starch granules in the conventional (AP only) and nominal (AP + boric acid) final adhesive compositions was noted. Formulations having uncooked starch in the hot (195-200°F) aqueous phase may slowly cook and gelatinize to produce a troublesome increase in viscosity on prolonged holding. Because no thinning agent (ammonium persulfate) is left, the cooking of the raw starch tends to produce a high molecular weight component which causes a high resultant viscosity increase. By adapting the concepts used in preparing "no carrier" adhesive for hot corrugating, it was thought possible to avoid this viscosity increase and, in addition, get a stronger adhesive with the added advantage of faster bond development.

Hot-corrugating "no carrier" adhesives are prepared by careful addition of a warm caustic soda solution to a slurry of raw starch under agitation. The caustic gradually swells the raw starch until boric acid is added to stop the swelling. The result is a suspension of swollen starch in an alkaline borax-containing aqueous system.

TABLE IX

ALKALI PRESWELL ADHESIVE FORMULATION

Slurry Composition (in order of addition)

Water	78 liters	(33% Starch solids)
50% NaOH solution	150 mL	
Stir 2-3 minutes		
Starch (as is)	100 pounds	
Stir 30 minutes	pH 10.7	
Boric acid (tech.)	40 grams	(0.1%)
Ammonium persulfate	123 grams	(0.3%)

By adding to the room temperature water the quantity of 50% sodium hydroxide normally used for post cooking addition to the starch slurry, a liquid of pH 10.5 to 11.0 was obtained as starter for the new adhesive formulation. Starch was then added (in a ratio of 100 pounds of starch to 78 liters of water) and the slurry stirred for about 30 minutes at temperatures around 70-75°F. During this time, starch granules swell enough to remain suspended without agitation, but not enough to gelatinize, or become sticky or gummy, or appreciably raise the slurry viscosity. After addition of AP and boric acid, the swollen, alkaline, starch slurry was cooked through the jet. Two variations of this formula were tested for increases in bond rate development. By the subjective bench test the bond development properties were rated excellent.

For this adhesive, as for the others, the cooked material was post-adjusted to about pH 9.0 with 50% sodium hydroxide.

The Brabender viscosity of this adhesive was 650 units, the "gel" temperature was 82°C (179.6°F), the solids 31.7% and the molecular weight of the adhesive polymer 67,031. This compares with a molecular weight of about 32-33,000 for the nonpreswollen adhesive.

d. Increased Solids, Alkali Preswollen Adhesive (Table X)

Statements in the literature on the nature of the final DF bond for hot corrugating suggest that the solids fraction of the adhesive line must be raised to a quite high level to develop enough bond strength to withstand the slitting operation at 600-650 fpm. Accordingly, it was postulated that increasing the adhesive solids level by adding clay would improve bond development. A formula for testing this idea is given below in Table X.

TABLE X

INCREASED SOLIDS, ALKALI PRESWELL ADHESIVE

Slurry Components (in order of addition)

Water	60.5 liters	(36.6% Starch solids)
Quadraphos (dispersant)	5 grams	
Engelhardt "Lamina" clay	10 pounds	(Bulk cost $0.042/lb)
50% NaOH solution	110 mL	
Stir to wet out clay		
Starch (as is)	100 pound	(about 10% water)
Stir 30 minutes to wet out and swell		
Ammonium persulfate	128 grams	(0.3% on starch)

The Brabender viscosity was 450 units and the "gel" temperature was 82°C (176°F). Bond development was rated by the subjective test as very good, but adding the clay did not improve upon the results obtained by preswelling alone.

e. Polyvinyl Alcohol as a Slurry added Adjunct (Tables XI, XII, XIII)

From the literature on coating binders and other sources, one is led to believe that polyvinyl alcohol, added to the starch slurry, would produce faster, stronger combined board bonding. Some of the exploratory experiments in which polyvinyl alcohol was included as a slurry additive are outlined below in Tables XI, XII, and XIII.

TABLE XI

SLURRY COMPOSITION

Water	66 liters	(36.6% Solids)
Elvanol 72-60[a]	110 grams	(0.27%)
Starch	100 pounds	(about 10% water)
Ammonium persulfate	128 grams	(0.3%)

[a]No longer commerically available. Elvanol HV current similar type.

The Brabender viscosity of this was 240 units, the gel temperature was 71°C (160°F), and the bond development was rated as good.

TABLE XII

SLURRY COMPOSITION

Water	66 liters	(36.6% Solids)
Elvanol 72-60	110 grams	(0.27%)
Starch (as is)	100 pounds	(about 10% water)
Ammonium persulfate	104 grams	(0.25%)

The Brabender viscosity of this adhesive was slightly higher (280) than the previous composition, with a gel temperature also slightly higher at 73.2°C (164°F). The bond development was also rated as good.

TABLE XIII

SLURRY COMPOSITION

Water	57 liters	(40% Solids)
Elvanol 72-60	110 grams	(0.27%)
Starch	100 pounds	(about 10% water)
Ammonium persulfate	128 grams	(0.3%)

The gel temperature was 68°C (154.4°F) and the Brabender viscosity was 240 units, initially. A second Brabender viscosity measurement, made sometime later, showed a value of 380 units. Upon being held an additional 20 minutes at 95°C, the viscosity had increased to 540 and was still rising when the test was discontinued. The marked, continuing rise in viscosity was believed to be due to continued cooking of the remaining raw starch in the hot, alkaline holding environment. When cooked in the absence of AP (exhausted during the normal jet-cooking process), the starch gives a large increase in viscosity.

Despite the difficulties in maintaining the properties of the adhesive in the holding tank, the subjective bond development ranking was rated as good.

f. Combining Polyvinyl Alcohol and Clay as Slurry-added Adhesive Adjuncts (Table XIV and XV)

This formula, Table XIV, combines the concepts of increasing solids by using clay, made up as a slurry like a coating slip, and adding dry starch and polyvinyl alcohol to this clay slip before cooking with ammonium persulfate.

After cooking, this adhesive was diluted with about 5 liters of water to reduce the viscosity to about 420 Brabender units. This dilution lowered the total solids level to about 56%. The "gel" temperature was 70°C (158°F). A subjective bonding rate assessment of very good was assigned to this adhesive.

TABLE XIV

SLURRY COMPOSITION

Water	34 liters	(About 59% total solids)
Calgon T (dispersant)	68 grams	
Engelhardt "Lamina" clay	50 pounds	
Starch	75 pounds	(about 10% water)
Elvanol 72-60	110 grams	(0.35% on starch)
Ammonium persulfate	96 grams	(0.3% on starch)

A summary of these and a few other adhesive formulations and their properties is given in Table XV. Although these adhesives showed some promise as faster bonding materials for achieving higher double backing speeds, neither the crude laboratory bond test nor the pilot system provided a good basis for quantitative evaluation. The lab test was simply too crude and too subjective. The pilot system was terribly expensive and time consuming, and could not provide the control and the data measurements necessary for the good evaluations needed for adhesive development. Faced with a critical need for effective bonding system analysis, and without suitable tools for the purpose, the project set out to design and construct the double backer simulator. This sophisticated but very efficient and workable test system proved of great value in subsequent bonding work.

9. Double Backer Simulator

a. System Description

The Double Backer Simulator was designed to provide a controlled, easily repeatable test for the relative evaluation of cold corrugating bonding systems. The full-scale double backing process was modelled as closely as possible to give direct significance to the test variables and test results. Time was chosen as the primary test variable because it is the time for bond setup that is the most critical parameter in a successful corrugating bond. In the simulator, a 2-inch wide by 12-inch long strip of single face board is bonded to a double face liner and the bonds are then broken one-at-a-time to create a measure of bond strength development with time.

TABLE XV

EXPLORATORY ADHESIVE FORMULAS

Formulation						Properites		
Starch Solids,	AP, %	Boric Acid, %	PVOH, %	Other Solids, %	Preparation	Visc.	GP, °C	Bond Rating
33	0.30	0.2	--	--	Standard	270	80	Good
41		0.2	--	--	Standard			Good
33	0.30	0.1	--	--	Alkali preswell	650	82	Excellent
36.6	0.30	--	--	Lamina clay 41%	Alkali preswell	450	82	Very good
36.6	0.30	--	0.27	--	Standard	240	71	Good
36.6	0.26		0.27	--	Standard	280	73.2	Good
40	0.30	--	0.27	--	Standard	240	68	Good
33.8	0.30	--	0.40	Lamina clay 25%	Standard	420	70	Very good
33	0.30	0.009	0.27	Citric acid 0.1	Standard	300	70	Good
36.6	0.30	--	0.27	Lamina clay 41%	Alkali preswell		80	Good
33	0.26	--	0.27	--	Standard	240	68	Good
33	0.30	0.2	0.27	--	Standard	270	80	Good

For proper simulation, the total time interval for the double backing process was divided into three segments: open time, or the time between glue application to the flute tips and the point where single face board and double face liner are joined; time under pressure, where the combined board is under the double backer belt; and the total bond time from initial contact until the board reaches the slitter-scorer. Because a running speed of 600 fpm could not be achieved in a laboratory instrument, simulation of these three time segments was accomplished by proper scaling of machine size and operating speed in conjunction with appropriate time controls.

The machine is modular in nature. The major elements can be seem in the overall view of Fig. 17. The machine frame includes a hot water system for temperature control (lower left), liner sample roll (lower center) and air control system (lower right). The top of the frame supports a jacketed adhesive pan and applicator system (left center), an air loaded combining section (center) and liner pull rolls (right center). Immediately below the liner pull rolls is a load cell system for measuring the bond strength. The single face sample rides in a carriage (upper left) supported by horizontal rods across the top of the machine. Also seen in Fig. 17 is the electrical control panel (above the machine) and the bond force recorder (extreme right).

The machine was designed to be portable. It rolls on casters and is compact enough to be transported in a van. Only air and electrical connections are required for operation.

For a test determination, the 2 x 12 inch single face samples were cut with the 12-inch dimension in the machine direction. This provided about 39 flutes for individual bond strength determination. The single face liner on the sample was bonded to a metal holder with two-sided tape. The holder with sample was slipped into the end of the sample carriage with the flute tips down and clamped in place with compression springs. The adhesive pan was prewarmed to the desired temperature by the hot water system and filled with adhesive. The desired adhesive film thickness was set on the adhesive pan applicator roll by adjusting the position of a single doctor blade. The applicator roll turned continuously at an idle speed. The double face liner was threaded from its supply roll over the air table combining section, around the load cell roll and then through the pull roll nip. A predetermined weight roll was suspended in a liner loop to provide proper liner tension.

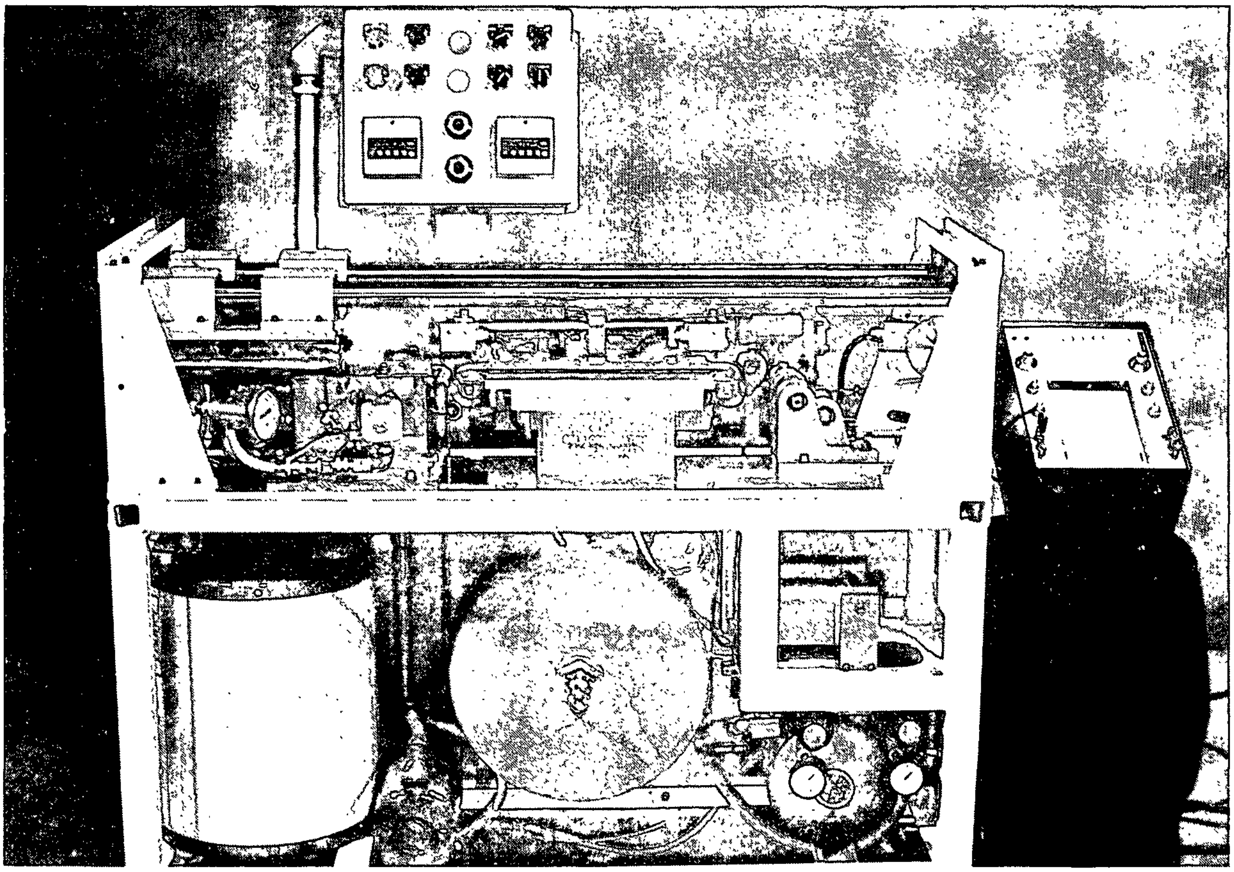


Figure 17. Overall view of double backer simulator.

During a test cycle, the sample carriage moves across the machine (left to right in Fig. 17) at a preset speed. The flute tips first contacted the glue roll to pick up adhesive. Carriage speed determined the open time between point of glue application and contact with the double face liner. The positional relationship between adhesive pan and liner can be seen in Fig. 18. At the start of a test, a clutch disengages the applicator roll from the idle motor; a second clutch engages to drive the glue roll synchronously with carriage speed. The double face liner also moves synchronously with the sample carriage.

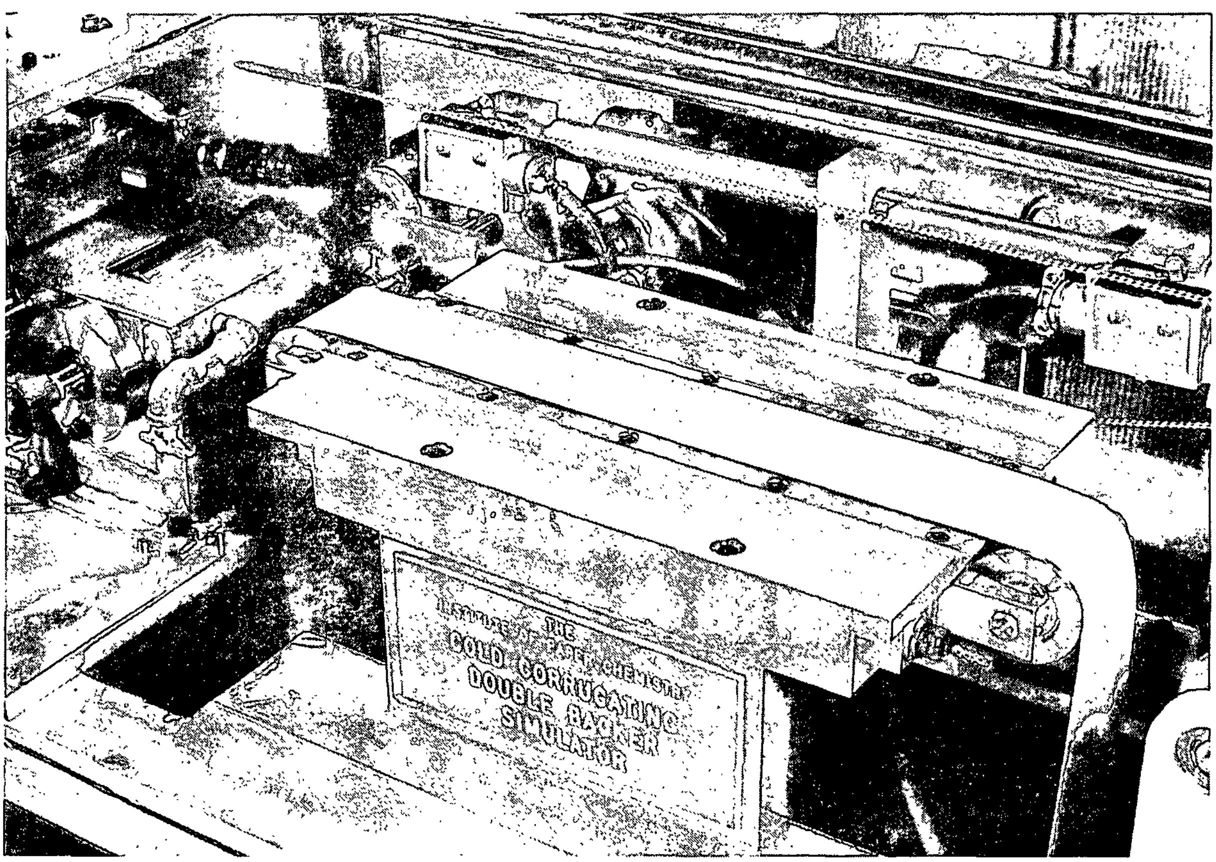


Figure 18. Adhesive application section and air table combining section of double backer simulator.

As the first flute of the single face sample contacts the double face liner, two control timers activate and air pressure is supplied to the air loaded combining section. The first 3 inches of the combining section are under the control of one pressure regulator, while the last 12 inches are controlled by a separate regulator. This allows the initial and the final segments to operate at different double backing pressures, if desired. A Teflon coated belt between the air supply ports and the double face liner distributes the air pressure and reduces drag. The sample carriage continues until the first flute reaches the end of the air table, at which time the carriage stops.

One of the two control timers determines the length of time that the air pressure is applied to the combined board to simulate the actual time under the top belt of a double backer. The second timer controls the time from bond formation until the start of bond breakage. When this timer deactivates, the sample carriage resumes forward motion at a predetermined speed (typically, one flute per second) to initiate bond breakage. As the carriage moves forward, the double face liner is moved along at the same speed by the liner pull rolls. However, the liner is pulled down over the end roll of the combining section and away from the single face sample (see Fig. 18, extreme right). This places the bond of each flute under tension as it passes over the end roll and causes it to fail. The tension in the liner increases as the carriage moves until the bond fails, at which point tension drops rapidly. This process is repeated for each flute. The load cell system measures this liner tension and, hence, bond strength. Bond strength is recorded as a function of time. The test cycle continues until all of the bonds are broken and then automatically stops.

A typical graph for a test cycle appears as Fig. 19. Bond strength appears on the vertical scale and bond age appears on the horizontal scale, increasing from left to right. At the bottom of the graph is an event line which drops slightly as the first flute contacts the double face liner. At this point, the air combining pressure is applied. Counting to the right from this point to any bond break at one second per major graph division determines the age of that particular bond. The event line returns to its normal position when the air pressure control timer deactivates. For each test, this identifies the amount of time that the combined board is under pressure. In the example of Fig. 19, the board was under pressure for 5-1/2 seconds.

As the test cycle begins (Fig. 19) the recorder first shows the liner tension induced by the suspended weight, usually about 2.0 pounds. As the cycle continues and the single face flute tips contact the glue roll and then the double face liner, the load cell output is irregular due to random variations in the system. After flute contact with the liner, tension decreases as bonds are formed to provide the necessary driving force. As additional flutes make contact, liner tension decreases to its lowest point.

In the bond breaking portion of the test cycle, the first few bonds exhibit low strength. As the cycle continues, however, the bond age and hence the bond strength increases as the adhesive continues the curing process. The presence of flute-to-flute irregularities, due in part to the high-lows, is evident. For this reason, single face samples are selected carefully and ten trial runs are averaged to give statistically reliable results.

Adhesives can be effectively compared with respect to rate of bond strength development and final bond strengths by plotting bond strength, as

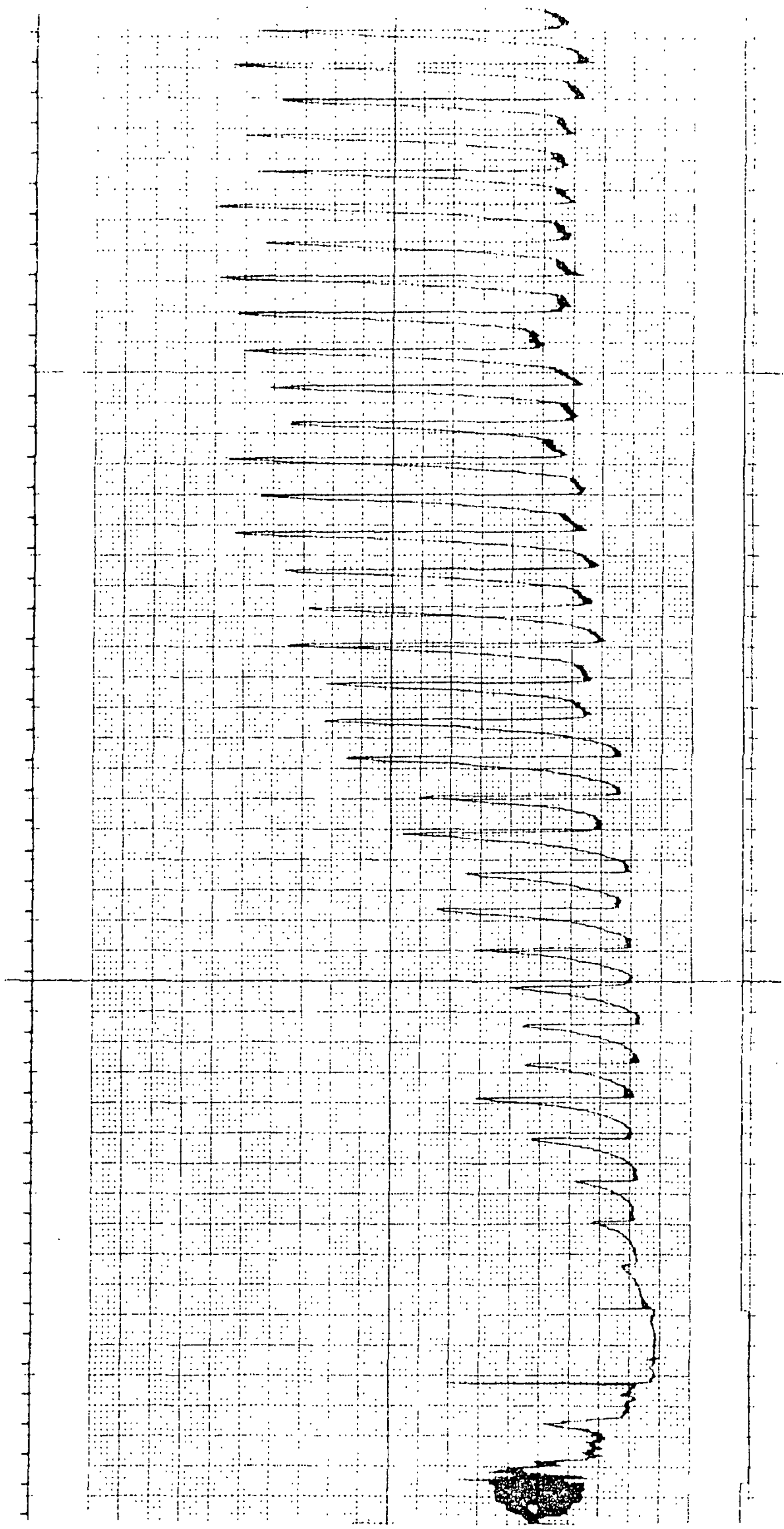


Figure 19. Double backer simulator chart.

determined from the peaks of the graph, as a function of bond age. Such a plot, along with the definitions and values of the parameters used to describe it, is shown in Fig. 20. Control adjustments on the simulator permit these comparisons to be made for any simulated speed of production, double backer length, combining pressure, glue application rate or type, component combination, etc. Thus, the double backer simulator provides an extremely efficient and versatile tool for the evaluation of bonding system factors.

b. Example Simulator Results

Most of the setback adhesive formulas evaluated over the past few years have much in common in terms of makeup, preparation, and properties. All started as pearl cornstarch slurries with ammonium persulfate as a modifier, all were jet cooked, and all were post adjusted in pH by the addition of 50% NaOH. Variations took the form of recipe proportions, other slurry additives, variations in cooking conditions, variations in final pH and other post cooking additives. For example purposes, a simple formula used frequently in pilot trials was selected as a baseline adhesive. Performance of this adhesive on both the pilot equipment and the simulator was known so that it could be used as a convenient reference for evaluating other adhesives from simulator data.

The baseline adhesive was formulated and prepared in accordance with the following recipe:

Slurry

Starch solids = 36% by weight

Ammonium persulfate = 0.3% on oven-dry solids by weight

Cooking conditions

285°F at 50 psig for 150 seconds

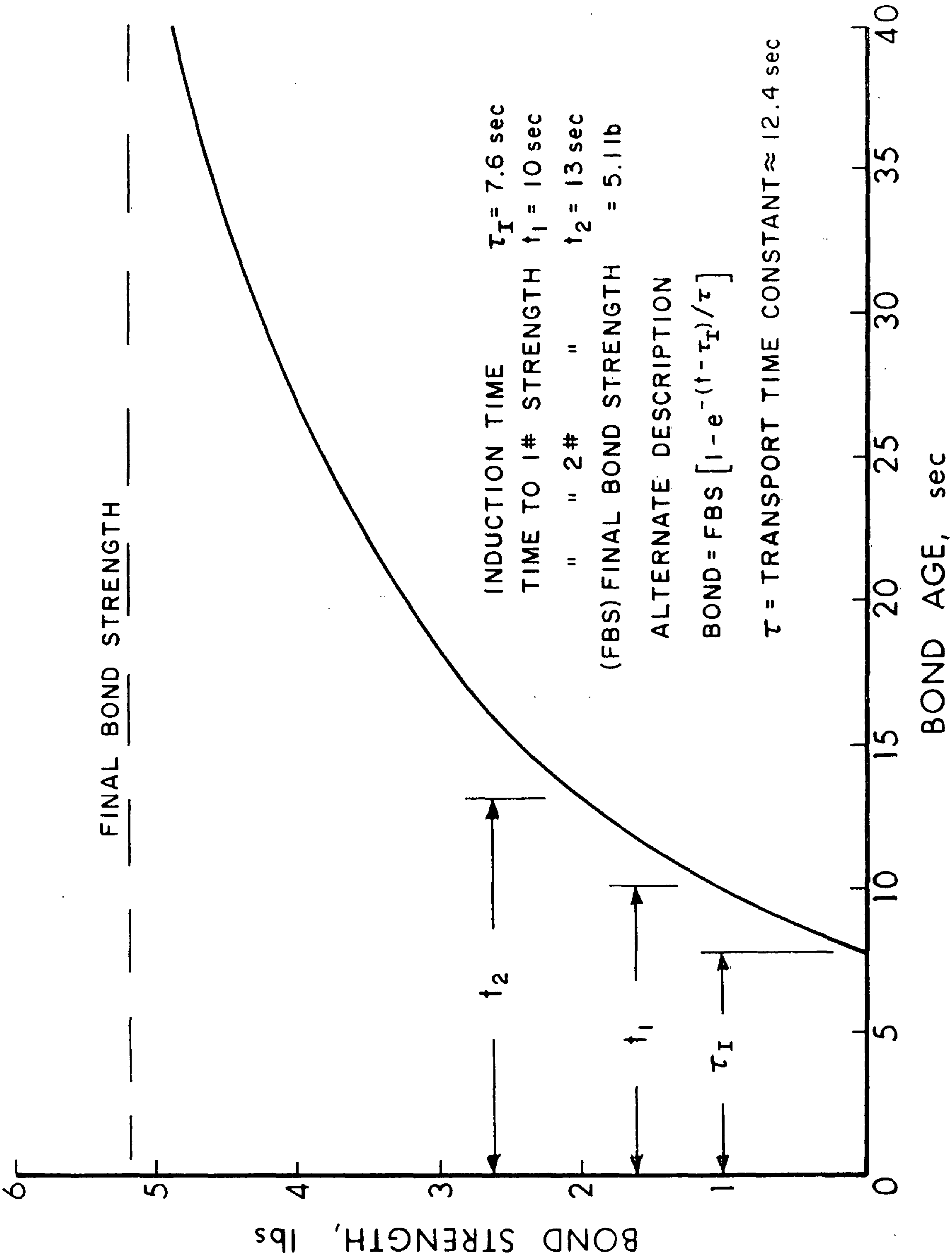


Figure 20. Typical simulator curve with parameter definitions.

Final pH and holding conditions

pH = 9.0

Holding temperature = 190°F

Adhesive properties

Starch solids = 32% by weight

Adhesive viscosity = 220 Brabender units = 385 cps

Typical bond strength data for this adhesive, obtained from the double backer simulator, is shown in Fig. 21.

When used as a trial adhesive on the laboratory single facer or on the pilot machine for the production of combined board, the baseline adhesive produced green bonds suitable for operation at 600 fpm. Final bond strengths were on the order of 5 lb/lineal inch, and most failures occurred within one of the components. Double backer production speed was limited, however, to speeds of about 300 fpm by the onset of slitter edge delamination. Above 300 fpm, the degree of loose edge was too great to be commercially acceptable. Hence, this baseline adhesive was not satisfactory for high-speed production of combined board, and other formulas with faster bonding were sought in subsequent work. Construction of the double backer simulator was prompted primarily by this goal.

c. Early Use of the Double Backer Simulator

Once assembled and checked out, the simulator was used to answer several holdover questions from the crude laboratory tests of various adhesive formulations. In particular, the ideas represented by the formulations listed in Table XV were tested without significant positive results, despite some of the earlier positive indicators from the subjective tests.

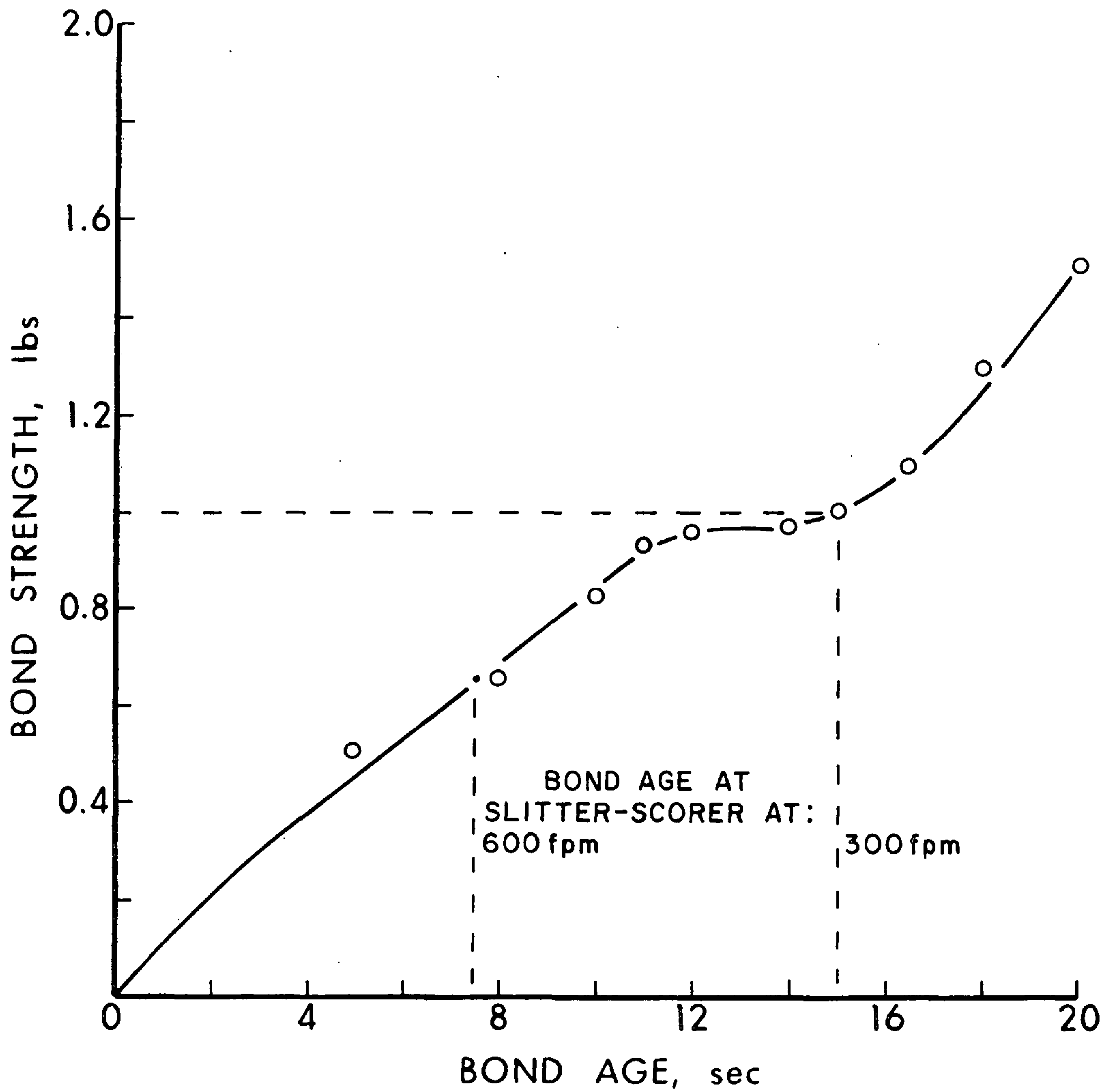


Figure 21. Bond strength vs. bond age for the baseline adhesive.

During this period, work continued on the generation of new formulas for test, but the simulator was used primarily to answer a number of questions about various factors in the bonding system. Examples include application rates and temperatures, open time, adhesive solids levels, liner preheating, conversion conditions, and so on. These investigations are discussed below.

During its early use the DBS was equipped with a smooth adhesive applicator roll. Film thickness on the roll and, hence, adhesive application rate was controlled by setting the gap between the roll and a trailing wiper blade. Later, the smooth roll was replaced with a gravure roll.

The simulator was an extremely productive tool and during a day of operation several different test conditions could be explored.

10. Effect of Adhesive Application Rate

As part of the effort to understand the fundamentals of double backer bonding, an evaluation of the impact of adhesive application rate on bonding was undertaken. This evaluation was conducted using the DBS. Some of the results are illustrated in Fig. 22, where bond strength is plotted against bond age for applicator roll film thicknesses of 2.5 and 12 mils. The corresponding application rates to the SF board were about 1.0 and 2.0 lb of starch/M ft^2. A typical 33% solids setback adhesive was used for these tests.

These data show that bond strength developed more rapidly as the application rate was decreased. For example, about 13 seconds were required to develop 1.0 pound of bond strength for the 2.0 lb application rate; only about 10 seconds were required when the application rate was reduced to 1.0 lb/MSF. The final bond strengths (pin adhesion values) were not significantly different for the two application rates.

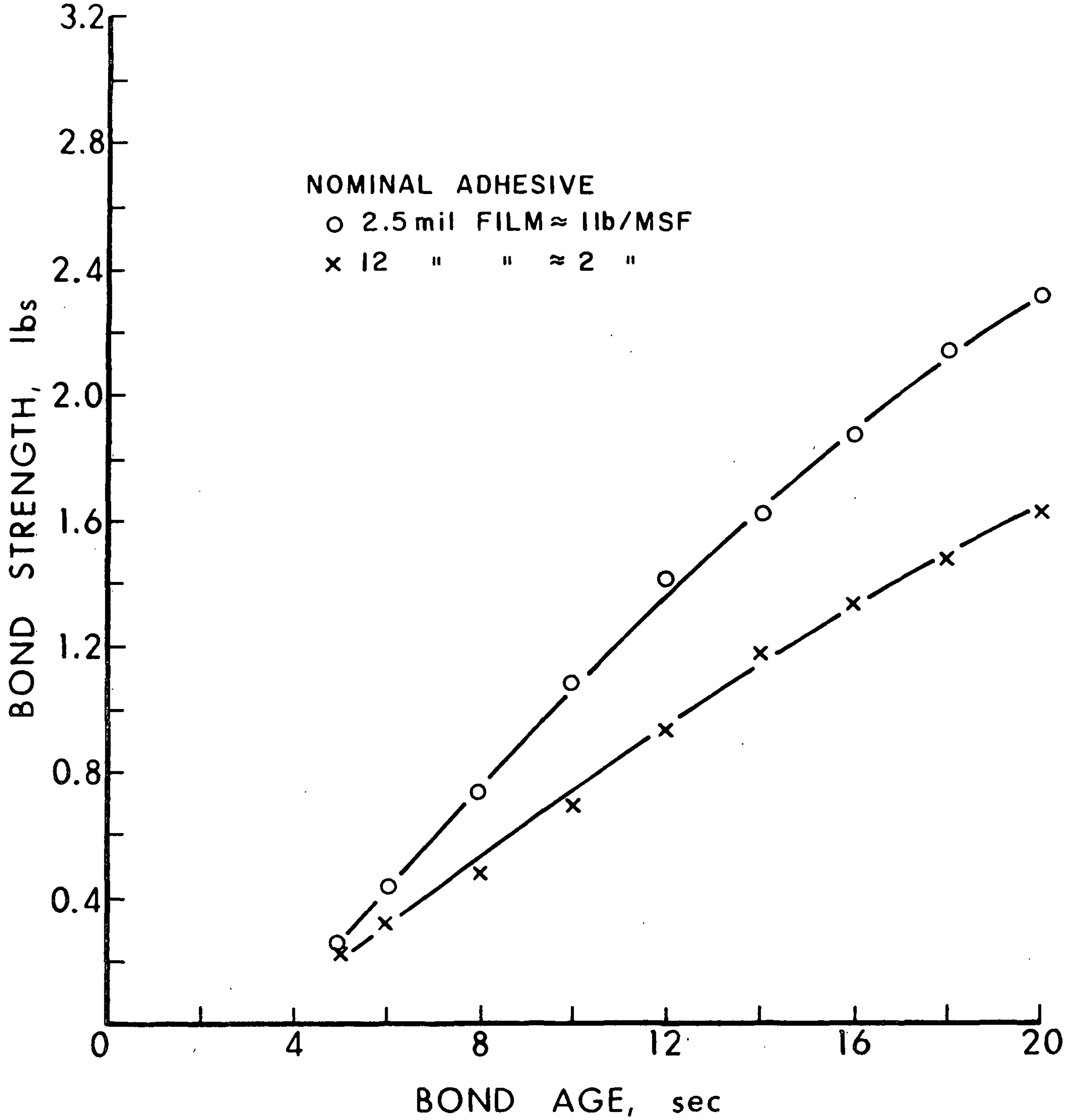


Figure 22. Effect of adhesive application rate on bond development.

11. Effect of Application Temperature and Open Time

As various other sections of this report show, there was an early belief that cooling was a dominant driving factor in the development of a green bond. Based on this belief, and in the absence of quantitative data, a special combining section was designed for the pilot machine to reduce open time and preclude premature cooling. Experiments on the double backer simulator and a simple transport analysis of the bonding process were later used to show that cooling is not a dominant factor in bonding. These data were sufficient to properly open time for machine design purposes.

12. Preliminary Heat and Mass Transfer Analyses

At the end of the open time period, the components are combined and heat and mass transfer processes become important. An evaluation of the heat transfer process during double backer bonding was conducted, assuming that heat and mass transfer (i.e., adhesive transport) are uncoupled. From this analysis, estimates of the temperature-time histories of the adhesive, medium, and liner were obtained.

Two aspects of the mass transfer process were evaluated; the effect of adhesive application temperature on the bonding process and the effect of the liner on adhesive transport.

a. Heat Transfer

Heat transfer during double backing occurs by two consecutive processes. The first process occurs between the glue station and the combining section of the double backer. In this process, hot adhesive is metered onto the flute tips at the glue station. As the flutes travel to the double backer, heat from the adhesive is conducted into the medium and convected to the ambient air, thus cooling the adhesive and heating the medium.

In the second process, the SF board is combined with the DF liner. Heat from the adhesive is transferred into the liner and medium. The DF combined board proceeds to thermal equilibrium as it travels to the slitter-scorer.

Both processes were modeled assuming one-dimensional heat transfer. The heat transferred into the trapped air spaces between flutes by convection and radiation is negligible and was therefore not included. Due to the poor thermal contact between the belt and the hot plates (slip sheets at the PCA pilot facility), the outside liner surfaces were treated as insulated. The following additional assumptions were made.

190°F adhesive (initial condition)

70°F medium and liner (initial condition)

80°F ambient air

C-flute, 26-lb medium, 42-lb liner

600 fpm corrugator speed

5 ft between glue and combining stations

Average adhesive film thicknesses: 2 mils applied to SF flute tips
1 mil between DF liner and SF flute tips

A transient heat transfer analysis of each process was undertaken using a finite-difference computer solution. The results indicate that within 0.5 second after the adhesive is applied to the flute tips (which is the time required for a flute to travel 5 ft at a speed of 600 fpm) the adhesive and the medium at the glue line have each reached a temperature of about 110°F. Thus, the adhesive has cooled by about 80°F and the medium at the glue line has increased in temperature by 40°F. Furthermore, the results indicate that 56% of the total heat is lost from the adhesive by convection to the ambient air and 44% is conducted into the medium.

The temperature distribution in the medium and adhesive at 0.5 second was used as the initial temperature distribution for analysis of the second process. The results indicate that 1.0 second after the DF liner and SF board are combined (i.e., 1.5 seconds after the glue has been applied to the flute tips), the medium at the glue line is at a temperature of about 93°F and the liner is at a temperature of about 86°F. After 2.0 seconds, the medium and DF liner are approximately in thermal equilibrium at 89°F.

The significant cooling of the adhesive between the glue and combining stations predicted from the theoretical analysis was crudely confirmed by a simple touch test of the adhesive film in a pilot trial. Thus, the double backer bond forms with an adhesive that is already cool. At first glance, this result suggested that the adhesive could be applied at a lower temperature with equivalent results. Later analysis and data will show that a high application temperature is important.

b. Mass Transfer

From the heat transfer analysis, it was concluded that bonding at the double backer takes place with cool adhesive. The necessity of hot application of the adhesive was therefore questioned. To answer this question, an exploratory adhesive was evaluated for bond development at application temperatures of 190, 180, and 170°F. The results of these tests, obtained from the double backer simulator (DBS), are presented in Fig. 23. These data show that the rate of bond development decreased as the application temperature decreased. For example, it is estimated that the maximum corrugating speed would be increased by 30-40% by a 20°F increase in application temperature from 170°F to 190°F.

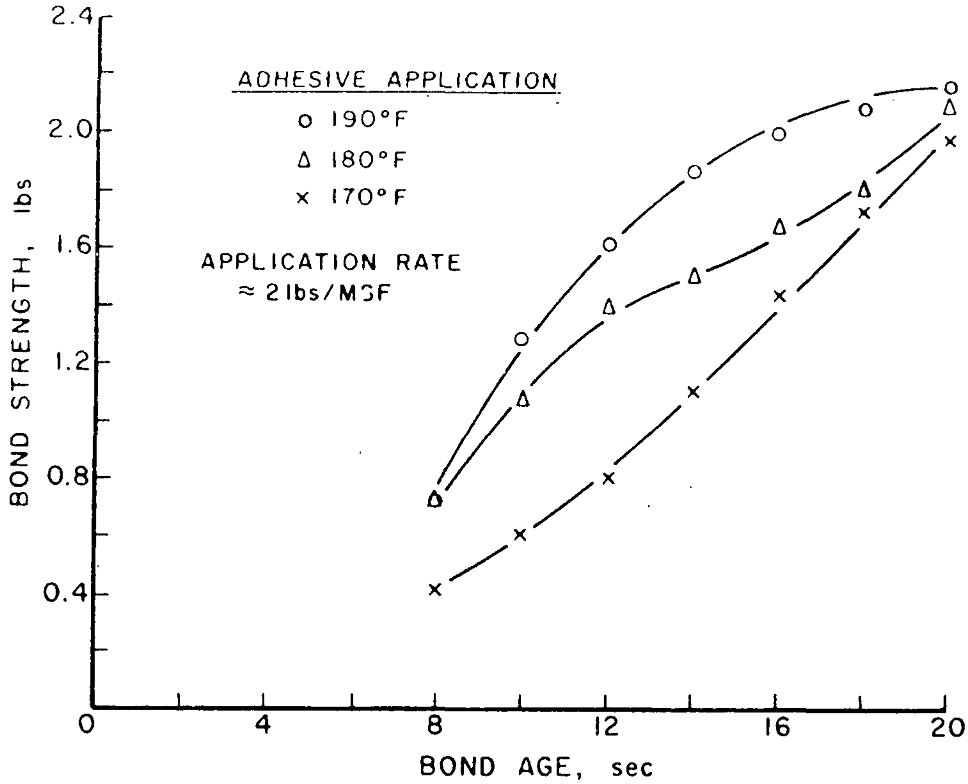


Figure 23. Effect of adhesive temperature on bond development.

The temperature of the adhesive during DF bonding will be approximately the same, about 110°F, for any of the three application temperatures evaluated. The time required to reach a specific bond strength increases as the adhesive application temperature is decreased; this implies a reduced rate of moisture loss to the medium and lines and a slower transfer of adhesive into the medium and lines.

The role of liner properties in controlling DF bonding was also investigated. Liners are significantly less receptive to water transport than mediums. This is demonstrated by the water drop numbers of liners which are usually one to two orders of magnitude greater than those for mediums. Thus, faster mass transfer of adhesive would be expected for a liner with a lower water drop number. To test this concept, a medium was bonded to SF board using the DBS. The medium selected had the same caliper (12 mils) as a normal liner, but a water drop value of only 22 seconds, compared to 600+ seconds for a normal liner.

The data in Fig. 24 illustrate the rate of bond development for SF board bonded to the medium. These data, shown for two different adhesive application rates, further illustrate the significance of this parameter to bond rate development. A comparison of medium to medium and medium to liner bond development is presented in Fig. 25. The medium to medium bonding process is much faster, but the ultimate bond strength is significantly lower than that of the medium to liner bond. These data clearly illustrate the importance of liner properties to fast bond development.

Based on the extremely rapid cooling process that takes place immediately after adhesive application, one would expect open time to be a relatively insignificant variable, so long as it remains within reason. This has been borne out by the lack of significant impact from the special combining section used in the pilot trials, from simulator data and from other sources. It would thus appear that normal corrugating machine gaps are suitable for cold corrugating.

13. Simulator Assessment/Bond Rate Factors

The simulator had proved to be a valuable and reliable tool for evaluating the DF bonding process, and from it some general understanding of the factors that control bond rate had been developed. Despite this confidence, it was desirable to know quantitatively the precision of the instrument and the effect

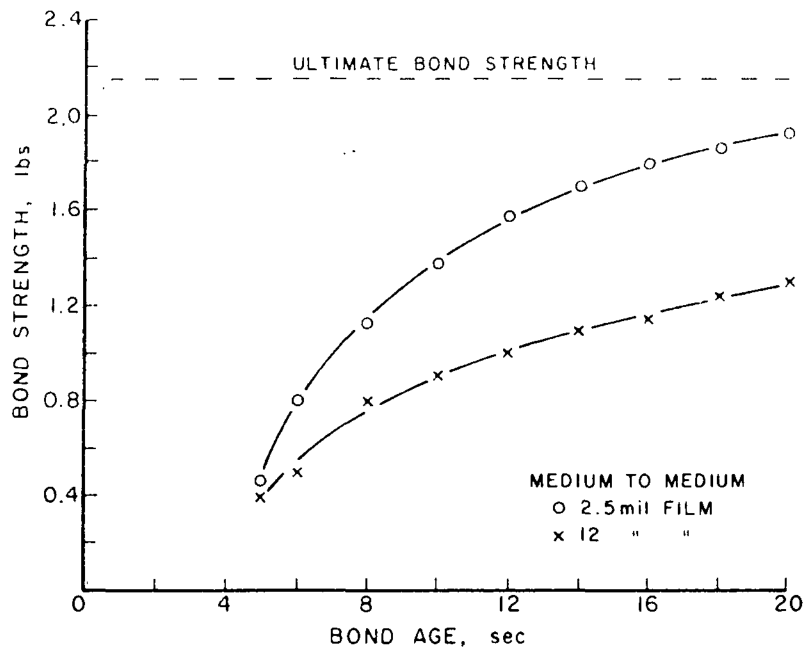


Figure 24. Medium to medium bond development.

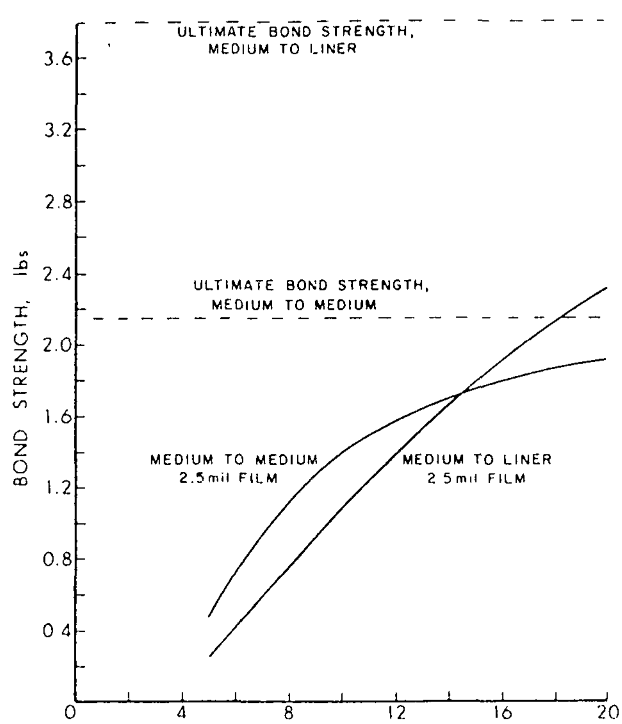


Figure 25. Comparison of medium-to medium and medium to liner bond development.

of changing certain variables. To accomplish this, the variation of adhesive properties was removed by selecting a shelf-stable material that remained "exactly" the same from day-to-day. A commercial polyvinyl acetate adhesive with bonding properties similar to those of the setback adhesive was combined with a factorial experiment design to yield useful information from a minimum of tests.

In a factorial design, each of the variables is allowed to assume either a low or a high value in any given test. By proper selection of the test pattern, a very few tests will reveal the influence of each of the variables and also the interactions between variables. A dummy variable is included to test the precision of the experiment. Variables for this test were chosen as follows: adhesive application temperature, carriage height (glue spread and combining pressure), adhesive application rate, combining pressure, single face moisture content, liner moisture content, and a dummy variable. Test results were taken as bond strength at 8, 12, and 16 seconds which corresponded, respectively, to speeds of 600, 450, and 300 fpm on the pilot machine. The test matrix and the results are shown in Table XVI.

Bond value changes little with the dummy variable, indicating that the precision of the experiment was quite good.

The influence of other variables on bond rate can be summarized in descending rank order as follows:

1. Adhesive application temperature: strong, positive
2. Medium moisture: strong, positive (dryer is faster)
3. Liner moisture: moderate, positive (dryer is faster)
4. Combining pressure: weak, positive (both pressure were quite high)

TABLE XVI

RESULTS FROM FACTORIAL EXPERIMENT WITH PVAc ADHESIVE

+ = Increase Bond - = Decrease Bond		Adhesive Temperature 150°F 100°F	Carriage Height -0.002 inch std.	Application Rate Higher Low	Table Pressure 10 psi 2 psi	Medium Moisture 35% RH[a] 80% RH	Liner Moisture 35% RH[a] 80% RH	Dummy	Results: Bond Strength at 8 sec	12 sec	16 sec
Run Number:	1	-	-	-	+	50%	50%		0.37	0.88	1.43
	2	-	-	-	-	-	-	-	0.15	0.27	0.45
	3	-	-	+	+	+	+	+	0.51	0.94	1.41
	4	-	+	+	+	-	+	-	0.20	0.44	0.79
	5	-	+	-	-	+	+	+	0.42	0.97	1.69
	6	+	+	+	-	+	-	-	0.74	1.38	2.05
	7	+	+	-	+	-	-	+	0.48	0.88	1.35
	8	+	-	-	+	+	+	-	1.33	2.11	2.84
	9	+	-	+	-	-	+	+	0.58	1.07	1.57
	10	+	-	-	+	50%	50%		1.44	2.23	2.87
Effects on:											
Bond at 8 sec		0.46	-0.24	-0.09	0.16	0.40	0.16	-0.11			
Bond at 12 sec		0.70	-0.18	-0.10	0.17	0.69	0.28	-0.08			
Bond at 16 sec		0.87	-0.10	-0.13	0.16	0.98	0.41	-0.03			

[a]35% RH - 5.5% moisture.
80% RH - 13% moisture.

5. Application rate: weak, negative (increased adhesive reduces bond)

6. Carriage height: moderate, negative (increasing tip compression reduces bond)

Liner moisture and adhesive application rate were both expected to be more influential. A starch adhesive may give different results, however.

These data served to increase faith in the simulator and understanding of the transport-limited bonding process. They also showed the factorial experiment to be an efficient and effective tool for bonding system analysis.

a. Evaluation of Setback Adhesives

Following a more or less classical experiment design, a large number of adhesive formulations was prepared and tested to examine the effect of formulation variables on bonding. These experiments were restricted to slurries made from pearl starch, ammonium persulfate, boric acid and water; to jet cooking under various conditions; and to post adjustment of the adhesive to pH 9.4 by in-line injection of NaOH. Primary formulation variables of interest included slurry solids level, ammonium persulfate level, and cooking temperature. Response variables of interest include adhesive viscosity and viscosity stability with temperature and time, and bonding properties as determined by the simulator. Interactions between adhesive properties and such combining environment variables as medium and liner moisture content are also important and will be discussed in the next section. The primary intent of this series of experiments was to examine the response of the adhesive to nominal combining conditions as an important step toward improving the adhesive.

In this experiment, all formulations that resulted in performance variables outside an acceptable range (e.g., high viscosity, poor stability, etc.) were discarded. Within the acceptable envelope, the best results were obtained by going to higher solids levels, higher AP levels, and higher cooking temperatures, all measured relative to those typically used in the past. Using a higher solids level reduces moisture added to the board and may yield an inherently higher adhesive strength. Higher solids also lead to higher viscosity, which is compensated by using more AP and a higher cooking temperature. The latter also improves adhesive stability.

The best adhesive from this series was made from a 42% solids slurry with 0.4% AP and 0.2% boric acid, all cooked at 150°C (302°F) to yield a viscosity of 350 BU. For comparison, most adhesives used in previous pilot trials were made from a 36% solids slurry with 0.3% AP and 0.2% boric acid, and cooked at 140°C (284°F) to yield a viscosity of about 280 BU. Although still higher solids might be useful, the dilatant nature of starch slurries precludes pumping and hence jet cooking at levels much above 42%.

A comparison of the bonding curves for these adhesives is shown in Fig. 26. It is clear from these data that increasing the solids level improves bond rate development. At a bond age of 8 seconds, for example, the time at which the board encounters the slitter in the pilot system for a production speed of 600 fpm, the high solids adhesive was about 25% stronger than the low solids adhesive. Final bond strengths for the two adhesives were comparable.

Two other adhesive properties of importance are adhesive viscosity stability with temperature and time. Both appeared to be improved by cooking at a higher temperature. Table XVII includes viscosity values for fresh and stored (2 hours) adhesives cooked at 140 and 150°C which show the greater stability produced by 150°C cooking. Table XVII also shows the effect of slight cooling,

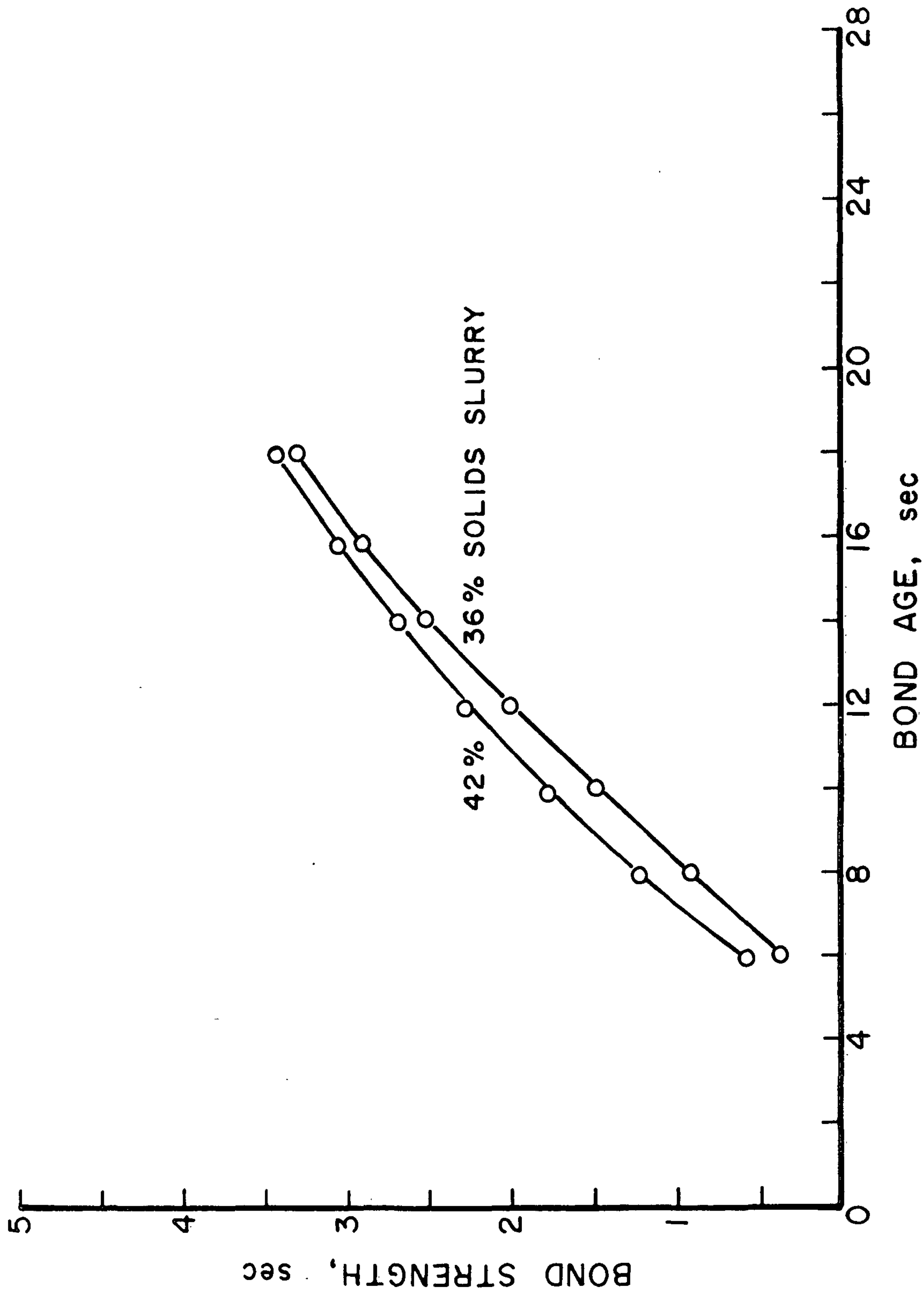


Figure 26. Effect of solids content on adhesive bond rate.

followed by reheating, on adhesive viscosity. Ideally, viscosity should increase with cooling and then fully recover upon reheating (at least for limited cooling) so that small temperature drops in the adhesive handling system could be tolerated. It is clear that the adhesive prepared at 150°C was better in this respect than the one prepared at 140°C.

TABLE XVII

VISCOSITY STABILITY WITH TIME AND TEMPERATURE

Cooking Temp., °C	Viscosity					
	Fresh 95°C	After 2-Hours Storage 95°C	Change, %	After Cooling to 75°C	After Reheating to 95°C	% Permanent Increase In Viscosity
140	330			770	510	50
150	215			540	250	16
140	385	425	10			
150	415	430	4			

b. Liner Preheating

It has been recognized from the outset that the DF bonding process is transport-limited and most severely so by transport into the liner. It has also been known for some time that a small increase in liner temperature greatly enhances the transport process and hence can be used to speed bond development. Thus, by adding a small amount of energy back into the process, much faster bonding could be achieved. This phenomenon has never been quantified, however. Such factors as the amount of benefit derived from a given temperature rise and the best mechanisms for supplying energy had not been explored. Limited early trials in the pilot system using the normal liner preheaters were inconclusive because of limitations in the steam supply.

To quantitatively study this process, the simulator was modified to permit liner heating. While a more thorough evaluation will be given in the next section, it is to be noted here that preheating has a dramatic positive impact on bond development rate. To illustrate, Fig. 27 shows the effect of liner preheating on the high solids adhesive. Note that the bond strength at 8 seconds is approximately doubled by preheating, in this case to about 150°F. Liner preheating thus offers one possible option for achieving full production speeds. The cost for doing so is about 10% of the energy now used in the hot process.

c. Bonding System Optimization

Bonding rate for the DF side is a function of many variables from at least four groups: adhesive properties, the adhesive applicator system, the combining system, and the components. Some of these variables have a strong influence on bonding and some interact in groups of two, three, or more. Some can be controlled wholly or partially by an operator, some must be set or at least bounded in equipment design, and some are beyond the control of either the designer or the operator. Enough has been learned about these complex relationships from past work to set design ranges and proceed with equipment development. As we move toward operation of this system, it becomes more important to explore the formulation/operation relationships and interactions to provide the basis for optimum operation of the system. Mapping of the adhesive response surface as a first step in this process was discussed earlier as was the factorial experiment with the PVAc adhesive to evaluate the nonadhesive variables. The next logical step is a combined simulator mapping of the bonding system comprised of the four subsystems.

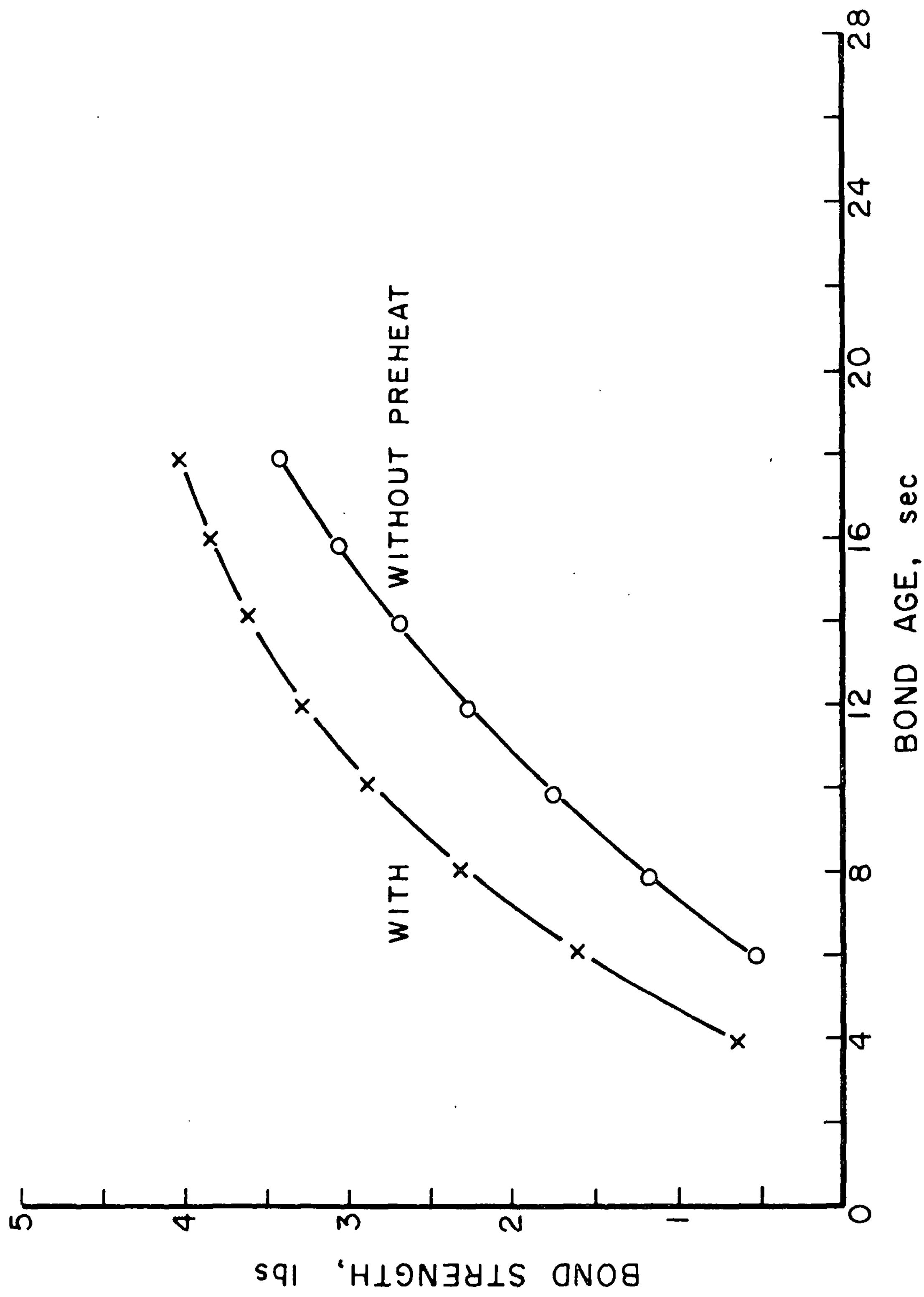


Figure 27. Adhesive bond rate with and without liner preheat.

The number and diversity of the variables and variable interactions in this mapping is staggering if viewed in the context of classical experimental design. Furthermore, the scatter inherent in the response of this system may preclude effective quantification of the relationships by a classical approach. For these compelling reasons, the factorial design approach has been taken with three primary objectives.

1. Discard the insignficant variables.
2. Identify the direction and strength of the single variable relationships.
3. Identify the direction and strength of any strong interactions.

Such an approach should provide enough information for approximate optimization in a given situation and for appropriate reaction to changing conditions. Of necessity, the numbers and ranges of variables explored must be limited to keep the scope of the experiment within bounds.

As a first step, the factorial experiment shown in Table XVIII was undertaken. Variables in this experiment included two identical adhesive cooks, liner temperature, adhesive application rate, combining pressure, medium (SF) moisture content, liner moisture content, and a dummy variable. Values selected for these variables are shown below. Results from the experiment are also shown in Table XVIII. The adhesive used for these experiments is that previously described, prepared from a 42% solids slurry cooked at 150°C.

Variable ranges for this experiment were as follows:

1. Adhesive preparation: two cooks on successive days

TABLE XVIII

RESULTS FROM SECOND FACTORIAL EXPERIMENT WITH HIGH SOLIDS SETBACK AHESIVE

	Cook	Application Rate	Medium Moisture	Liner Moisture	Liner Temperature		Table Pressure	Results: Bond Strength at		
+ = Increase Bond	No. 2	1.0 lbs/MSF	4%	4%	Hot		1.5 psi			
- = Decrease Bond	No. 1	0.5 lbs/MSF	9%	9%	Cold	Dummy	0.5 psi	8 sec	10 sec	12 sec
Run Number: 1	-	-	50	50	-		10	0.86	1.37	1.79
2	-	-	-	-	-	-	-	1.08	1.37	1.63
3	-	-	+	+	+	-	+	1.82	2.33	2.75
4	-	+	+	+	-	+	-	0.72	1.21	1.64
5	-	+	-	-	+	+	+	1.37	2.09	2.65
6	+	-	50	50	-		10	1.32	1.61	1.88
7	+	+	+	-	+	-	-	1.47	2.10	2.62
8	+	+	-	+	-	-	+	1.49	1.94	2.34
9	+	-	-	+	+	+	-	1.94	2.70	3.28
10	+	-	+	-	-	+	+	0.68	1.01	1.33
Effects on:										
Bond at 8 sec	0.15	-0.12	-0.30	0.34	0.66	-0.29	0.04			
Bond at 12 sec	0.19	-0.02	-0.36	0.40	0.75	-0.18	0			
Bond at 16 sec	0.22	-0.06	-0.39	0.44	1.09	-0.11	-0.02			

2. Liner temperature: 73 and 150°F
3. Application rate: ≈ 0.5 lb/MSF and 1.0 lb/MSF
4. Combining pressure: ≈ 0.5 psi and 1.5 psi
5. Medium moisture: ≈ 4 and 9%
6. Liner moisture: ≈ 4 and 9%

Salient results from these experiments were as follows:

1. The variations due to the two adhesive cooks were small, thus showing reasonable day-to-day repeatability in adhesive preparation.
2. There was more scatter (less precision) in this experiment than in the previous one with the commercial adhesive.
3. Liner temperature was a strong positive factor.
4. Medium moisture content showed a fairly strong trend, but in a direction opposite to that expected (wet components gave faster bonding).
5. Liner moisture content was also a fairly strong factor with the dryer liner being better.
6. Bond rate increased slightly with increased application rate (≈ 0.5 lb/MSF to ≈ 1.0 lb/MSF). Again, this was opposite to the direction expected and may be a reflection of higher inherent strength in this adhesive, of the higher solids level, or an interaction artifact from liner heating. In this range, application rate may not be an important factor. At higher rates, it is critical.

7. Bond rate was a positive but fairly weak function of combining pressure.

Some interaction effects not tabulated but possibly important to understanding the bonding process were as follows:

1. Less adhesive was better on a hot liner; more was better on a cold liner.
2. Medium moisture was not important with a hot liner; a moist medium was better with a cold liner.
3. Less adhesive was better with low combining pressures.

Taken together, 1 and 2 suggest that the adhesive line must be kept wet, and hence fluid, until the liner transport process begins. Optimum bond rate development thus requires proper balance among medium moisture content (single face adhesive application rate), DF adhesive application rate, and liner conditions (e.g., liner temperature). The results of these experiments provide the basis for this balancing process. They also support applying the adhesive to the liner first, although the practical problems of doing so are real.

These data are all for adhesives with a solids content of about 39% as compared to 33% in most previous experiments. The results show reduced sensitivies to adhesive application rate and medium moisture and perhaps to liner temperature as well, although this has never before been evaluated. Based on these data, two variables deserving of renewed attention are open time and application temperature.

d. Board Grade Effects

Bonding in the cold corrugating process depends on moisture transport to the medium and liners. This moisture must be absorbed by the components during the bonding process and then slowly given up to the surroundings in the stack. The effects of board grade (liner basis weight) on moisture transport rate (and hence bond development rate) and final moisture content are both important.

Simulator tests with 26- and 42-lb/MSF liners, Fig. 28, show no significant difference in bonding rate, thus suggesting that basis weight is not important during the bonding transient. Basis weight is important, however, in determining the equilibrium moisture content of combined board, but not as important as adhesive solids fraction and application rate. This is illustrated by the curves in Fig. 29 which show moisture added (by the adhesive) as a function of adhesive application rate. Curves are shown for solids fractions of 33% (standard in the past), 39% (now being used), and 50% (a desirable target) and for two board grades, 125 psi, and 200 psi. The trends for still heavier grades are evident. Note that changing solids from 33 to 39%, a six percentage point increase, gives a large reduction in moisture added, thus illustrating the importance of maximizing solids content.

e. Futher Exploration of Application Rate Effects

In all previous tests with the double backer simulator a smooth adhesive applicator roll was used. Application rate was controlled by the gap between a metering blade and the roll. Some early experiments showed the importance of application rate to the rate of bond development. Application with smooth rolls, however, tends to position the adhesive on the "shoulder" of the flute tip, leaving a "dry" centerline at the very tip.

To further explore the application rate and distribution issue, three applicator rolls were tested: a smooth roll, a 16 quad gravure cell roll and a

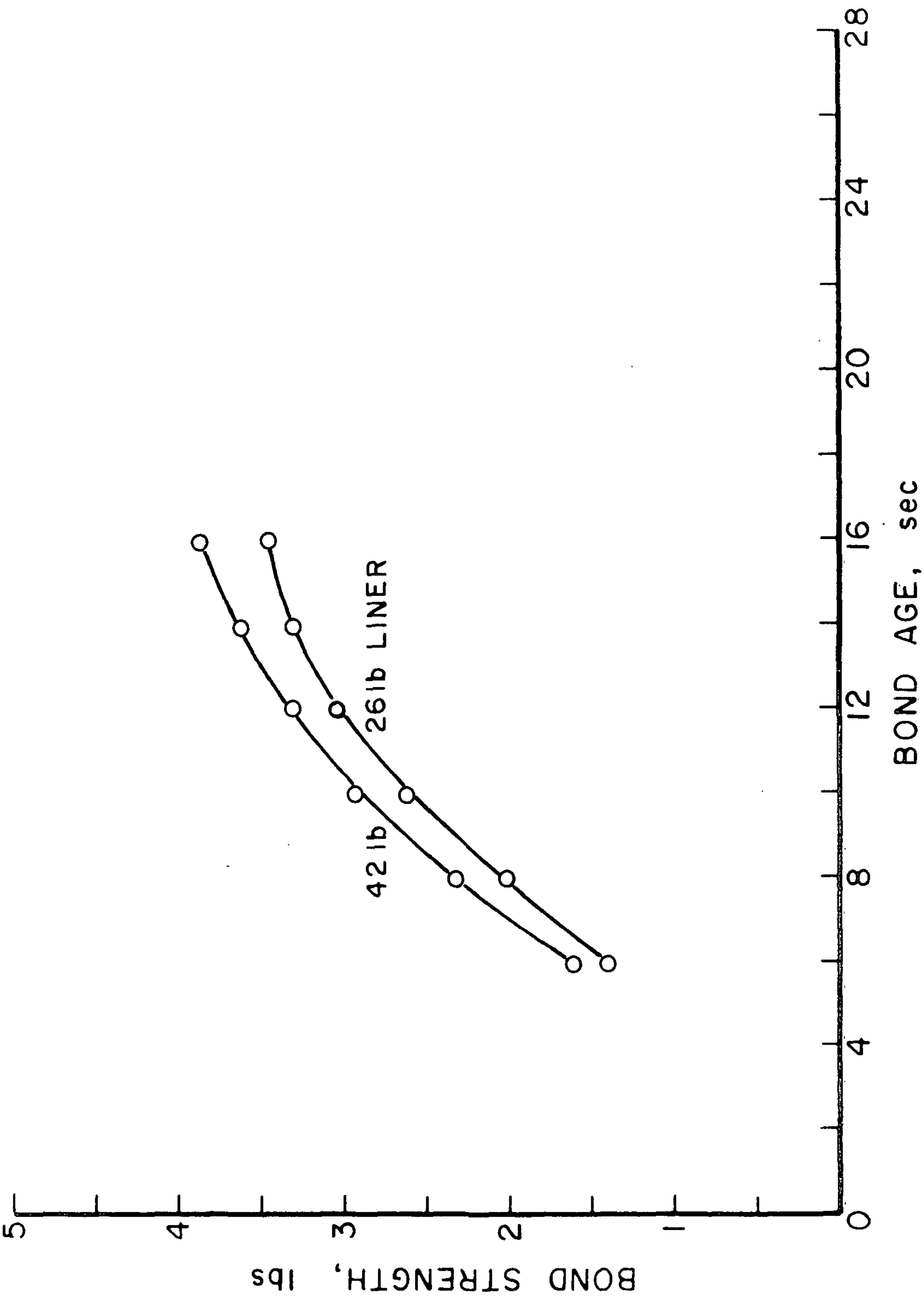


Figure 28. Relative bonding rates for 26- and 42-lb liners.

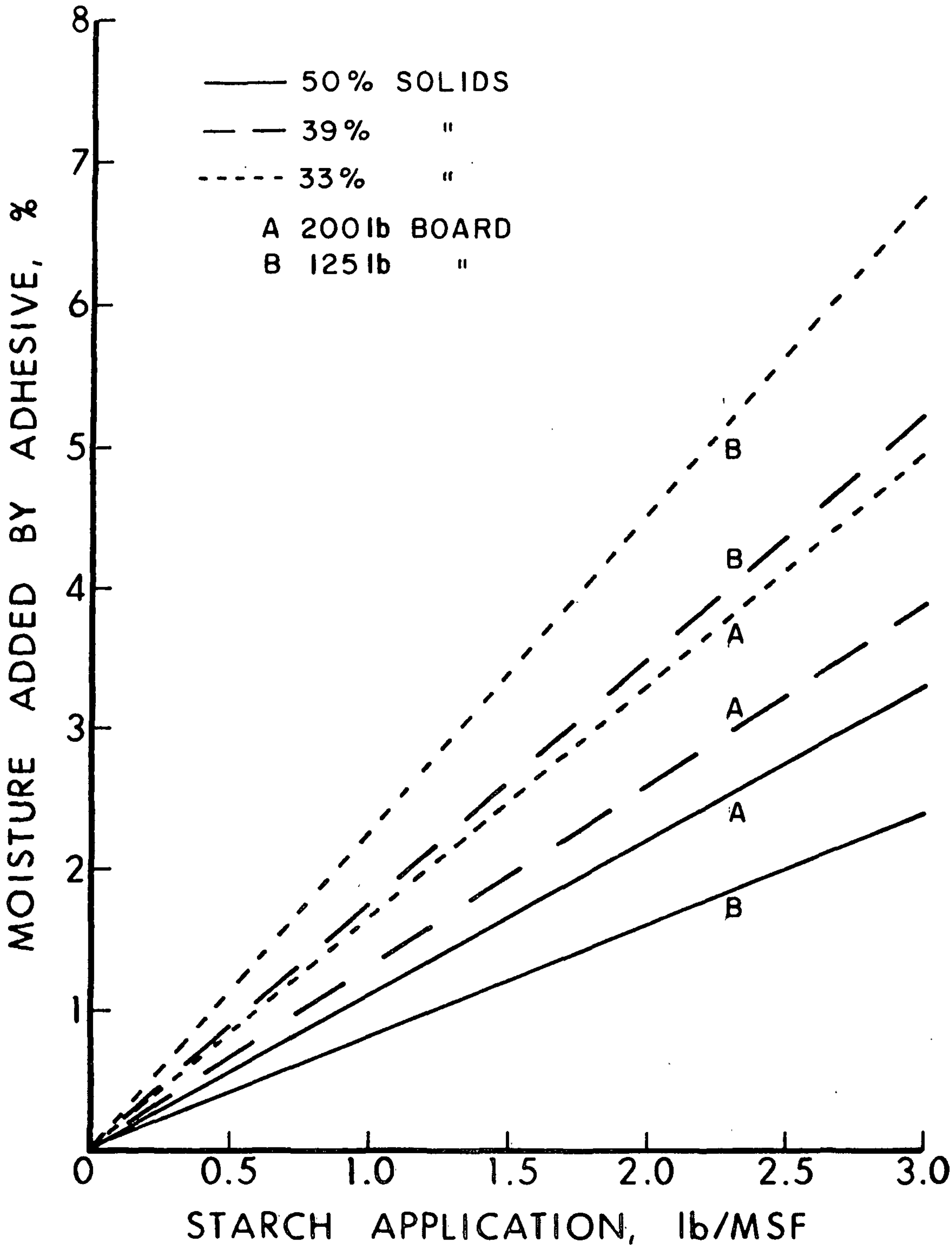


Figure 29. Moisture addition by the adhesive for various grades and solids levels.

25 quad roll. Various application rates were obtained with each by controlling the surface film thickness.

Data from these tests showed that (1) pin adhesion values for a given application rate were a weak function of roll pattern and (2) pin adhesion levels were acceptable in level and modestly related to application rate over the range from 0.5-1.75 lb/MSF. In contrast, bonding rate data favored use of low application rates and a gravure roll. Based on these results, a 25 quad gravure roll, wiped clean by a trailing wiper blade, was used for all subsequent studies. This combination applies about 0.5 lb/MSF of starch under typical operating conditions. The starch is applied in a very narrow, thin line on the center of the flute. The photomicrograph in Fig. 30 shows a section of a bond formed from this system. Concentrations of the adhesive along the bond line and the effectiveness of the bond are evident in this figure.

f. Further Exploration of Open Time

Using the 39% solids adhesive and the 25 quad applicator roll, the effect of open time on bond development was reassessed. These experiments showed that open time is not important unless it exceeds about 1.5 seconds. Hence, open time is not a critical issue for machine design.

g. Translation of Simulator Results to Pilot Trials

As they were obtained, simulator results were used to shape the conditions for pilot trials. Most notable among the important contributions to the pilot trials were higher solids, higher cooking temperature, lower application rates and higher combining pressure. The pilot trials results for all of these changes are described in the pilot trials section. Availability of the simulator at an early date - 1977 - would have done much to speed progress, reduce costs and improve the results from the pilot trials or discourage their undertaking altogether.

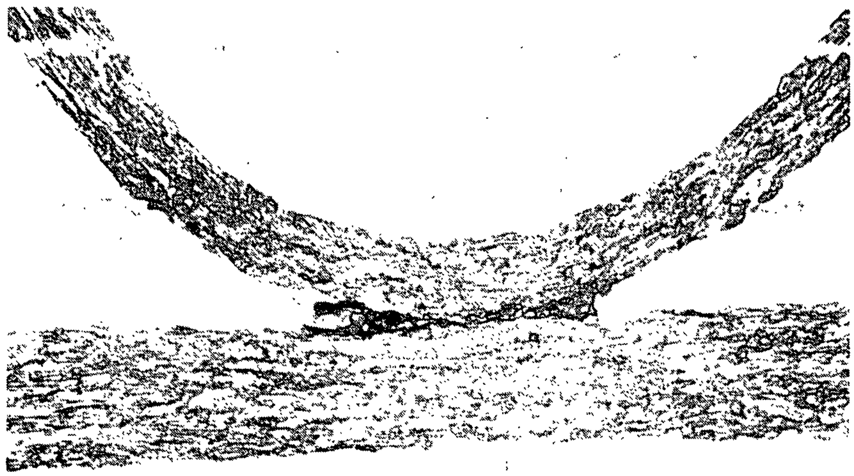


Figure 30. Photomicrograph of bond formed with 25 quad gravure roll (0.5 lb/MSF application rate).

14. Further Adhesive Developments

Cold corrugating requires an inexpensive adhesive that develops proper bonds with little or no heating of the components. Early work on such adhesives, initiated in the early 70's, identified thermochemically modified pearl starches as prime candidates for this application. Concerted efforts at developing this adhesive, started in the mid-70's, used very crude and simple cookers and devoted attention primarily to single face bonding. Pilot work, started in 1978, provided the first evidence of the double face bonding properties of the modified pearl starch adhesives. It was immediately clear that the DF bond requirements were very demanding, perhaps more so than the SF bond requirements.

Over the intervening years much effort was devoted to developing workable adhesives with rapid bonding properties for the DF bond. Along the way, the double backer simulator was developed as a sophisticated tool for quantitative evaluation of the DF bonding process. Most of the adhesive formulation work was empirical and experimental in nature and was directed to the chemical aspects of the conversion process and the finished adhesive. Simulator results were used to guide setting of other combining parameters to yield the best overall performance of the bonding system for pilot trials.

As a result of that work, a bonding system was developed to produce acceptable single face board at 600 fpm without any heating of the paperboard components. Similar speeds were obtained occasionally on the simply converted pilot double backer, but only by using DF liner preheaters and special techniques to increase combining pressure. These explorations left few untapped ideas with respect to basic adhesive formulation, thus requiring that future work move toward more dramatic variations in the adhesive and to better understanding of the bonding transport processes. Some of the areas explored and the results obtained are outlined briefly below.

a. Conversion Conditions

The major cooking variables (slurry solids, AP, cooking temperature, pH) and their relation to bonding rate and operating parameters have all been studied extensively. Best bonding is generally obtained at the highest workable solids (about 39%), at conversion levels permitting good flow in the applicator system (viscosities of about 300-400 BU) and at cooking intensities which permit full dispersion of the adhesive.

b. Molecular Weight Distributions

In a further effort to improve bonding, attempts were made to modify the molecular shape and size distribution of the starch in the adhesive. In general for starch pastes, higher molecular weight adhesives give stronger bonds; higher solids require lower molecular weights for workable viscosities. A highly modified starch containing a portion of high molecular weight starch should give improved strength while still retaining good fluidity and rapid bonding.

Such a hybrid starch adhesive was produced in two ways. One was prepared by the technique of AP holdout, a method of modifying a portion of the starch in an adhesive preparation differently from the rest. In this method, adhesive preparation was started with very little AP in the slurry to produce a portion of adhesive with limited conversion. After a selected time interval, the portion of AP "held out" was added to* the remainder of the slurry for normal conversion of the remainder of the starch. In a second method, pearl starch was combined with a highly modified dextrin before cooking. This combination has a bimodal molecular weight distribution that is partially retained in the final adhesive. Typical results from this work are shown in Table XIX. Generally, no significant improvements in bonding rate were found.

A similar effort to improve adhesive performance by modifying the molecular composition was made by varying the amylose to amylopectin ratio. Starches high in amylose or amylopectin were used in combination with pearl starch. The results are shown in Table XIX. Although gains in bond rate were sometimes

*This is an approximation of what is believed to have happened in the October, 1978, trial which produced excellent bonding results, but a generally unmanageable adhesive.

observed with amylopectin blends, straight amylopectin gave poorer bond rates. Increasing the amylose content did not strongly affect the bonding rate.

TABLE XIX

MODIFICATION OF STARCH MOLECULAR SIZE AND SHAPE DISTRIBUTION

Starch	AP, %	Slurry Solids, %	Cook Temp., °C	Visc., BU	Bond Strength		Pin Adhesion
					8 sec.	10 sec.	
Pearl	0.4	41.8	150	330	1.14	1.70	76
Pearl	0.4[a]	41.6	150	330	1.37	1.73	77
Pearl	0.3	35.7	141	310	0.89	1.37	77
1/3 Pearl 2/3 High Fluidity	0.1[b]	36.3	140	430	0.80	1.17	88
Pearl	0.4	41.8	150	360	0.93	1.49	79
3/4 Pearl 1/4 Amy-lopection	0.3[c]	41.6	150	395	1.17	1.68	83

[a]AP held out until about 10% of starch was cooked.
[b]AP equivalent to 0.3% on pearl starch.
[c]AP equivalent to 0.4% on pearl starch.

c. Cross-linking

Several other modifications to the adhesive formula were made in an effort to improve bonding. Addition of the cross-linking agent, glyoxal, gave better pin tests but poorer bond rates. The addition of glycol to the final adhesive to act as a wetting agent did not significantly improve the bonding rate. Addition of the more powerful wetting agent, Surfynol, reduced the bonding rate and, perhaps, the pin adhesions, too.

d. Grafting

Modification of the starch by graft copolymerization of itaconic and acrylic acid during cooking gave improvements in bond rate but the results were confounded by a sharp increase in viscosity. Figure 31 shows that a lower solids, grafted adhesive gave almost the same bond as ungrafted adhesive at high solids. A grafted adhesive at high solids showed improved bond rate, but at considerably increased viscosity.

e. Liner Treatments

On the basis of studies of the operating variables for the Double Backer Simulator, several efforts were made to improve bond rate by pretreating the linerboard.

Earlier work indicated that increasing liner moisture reduced bonding rate, while heating the liner increased it. These results were reaffirmed in the laboratory and in pilot trials. Mild chemical modification of the liner surface was attempted by treating it with various dilute solutions and drying. All of these treatments caused some improvement in bond rate. However, the same treatment using distilled water caused a similar improvement. The nature of the chemical did not appear to affect the bonding rate signficantly even though it did affect the surface sizing of the liner. The application of heat to the treated liner before bond formation improved the bond rate over that of the unheated, treated liner (Fig. 32), but heat by itself did not produce a permanent change in the liner that would improve bonding rate.

f. Conclusions

All of this information led to several conclusions. The bond formation system is limited by the transfer of adhesive onto and into the board components. Significant gains in bond rate will be made on the basis of a better understanding of the mass transfer phenomena involved in the formation and setting of the adhesive bonds.

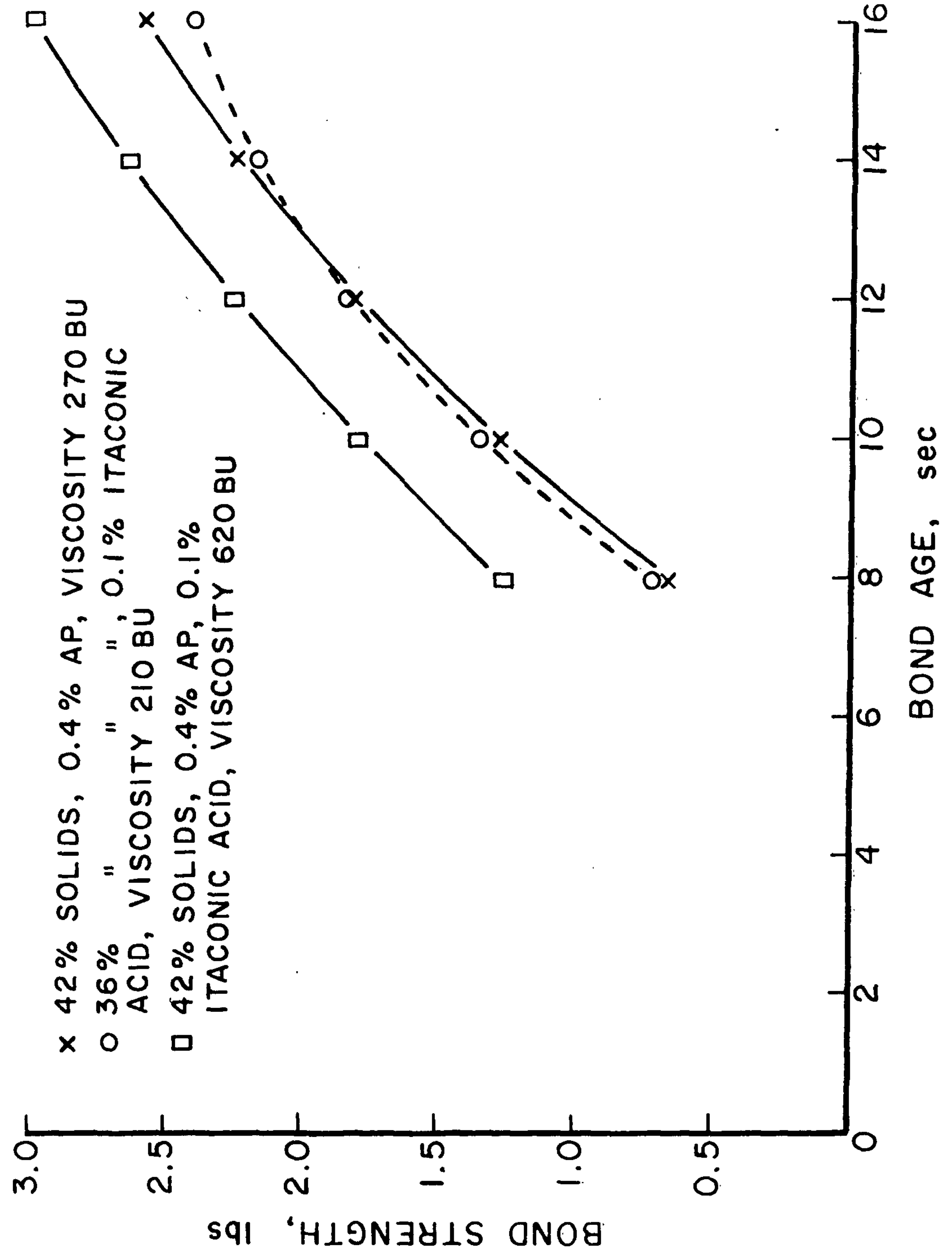


Figure 31. Effect of itaconic acid grafts on bond rate.

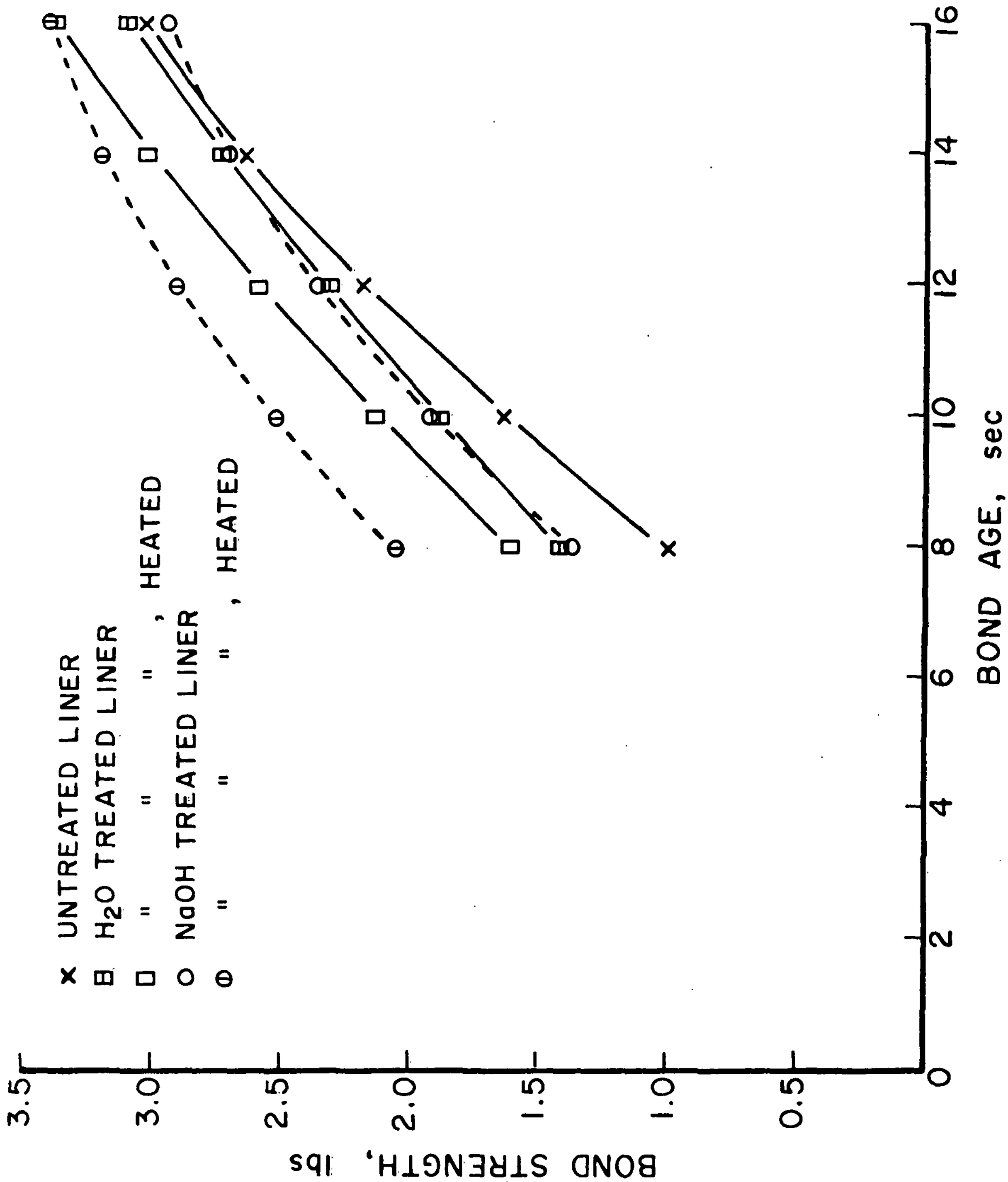


Figure 32. Effects of liner treatment and liner heating on bond rate.

The bond rate gains from increased solids have reached a limit because of the constraint of incipient dilatancy in the starch slurry. The high molecular weight desirable for bond strength has been balanced against the reduction in molecular weight needed to attain good, workable viscosity. At the same time, conversion conditions have been selected to give a thoroughly cooked starch for good adhesive stability. No real improvements were noted with the attempts to change the molecular size and shape distribution of the starch adhesive. While some gains may have been made from graft modification, the results were not clear and gains appeared limited.

The positive effect of heating the liner on the bond rate implies that the process is limited by transfer phenomena. The effect of treatment of the liner with dilute solutions also implies a transfer limit. The effect is not chemical. Distilled water is essentially equivalent to dilute sodium hydroxide. It is not a matter of surface sizing. The hydroxide-treated liner has lost its surface sizing; the water-treated liner has not, yet the bonds are about alike. The one difference observed in the treated liner is a very slight disruption of the surface fibers, visible under the microscope at low magnification. The effect of liner treatment on the bond strength vs. bond age curve appears to raise the curve somewhat preferentially at the shorter times and thus to reduce the induction period. The effect of heating the liner is to raise the curve more or less uniformly (Fig. 32).

In the final analysis, bond development appears to be by drying of the adhesive, that is, by mass transfer of water from the bonding adhesive. This is preceded by an induction period during which full contact is made and the transport gradients are established. The results of the empirical studies with the simulator form the basis for more detailed study of these transport processes. Further improvement in bonding rate will come from increased understanding of these phenomena or from fundamentally different approaches to the adhesive system.

15. Water Resistant Adhesives

As pointed out in the adhesive requirements section, a water resistant cold corrugating adhesive is a necessary part of a complete system, so much so that the development of a workable water resistant adhesive was a part of the definition of project success in the UCC/IPC subcontract. Some preliminary work of only limited success was described earlier in this section. The work described in this section was very limited in scope, but was successful in producing some measure of water resistance.

Because of the limited scope, all the work was carried out by forming water resistant samples of board on the double backer simulator. None of the trial adhesives was ever tested as a water resistant adhesive on either the laboratory single facer or the pilot machine.

a. Development of a Wet-Strength Test Fixture

Standard "thumb flick" tests, wet burst, and wet pin adhesion tests were found to be unsatisfactory for quantitative laboratory work. The thumb test is totally subjective and not quantitative. Wet burst is related more to the wet strength properties of the components than to bond properties and, furthermore, burst values are subject to inherent wide variability. Finally, the wet pin adhesion test is often influenced significantly by disruption of the bonds during pin insertion into a wet sample.

To provide a proper, controllable, quantitative measure of wet bond strength, a wet shear test was adapted. For this test, a sample of board 2 inches wide and 10 flutes in length was used. Extensions on the opposite liners were used to clamp the sample so it could be subjected to a machine direction shear load. To avoid handling the samples while they were wet, a special fixture for holding six samples was built. Samples were clamped in the fixture in the dry state and the complete fixture was then immersed in water for 24 hours; after soaking, the samples were drained, the fixture clamped in the Instron, and the samples tested one at a time. The fixture worked very well and avoided the necessity of handling the samples wet. A schematic of the sample holding and testing fixture is shown in Fig. 33. A picture of the multiple sample holder is shown in Fig. 1.

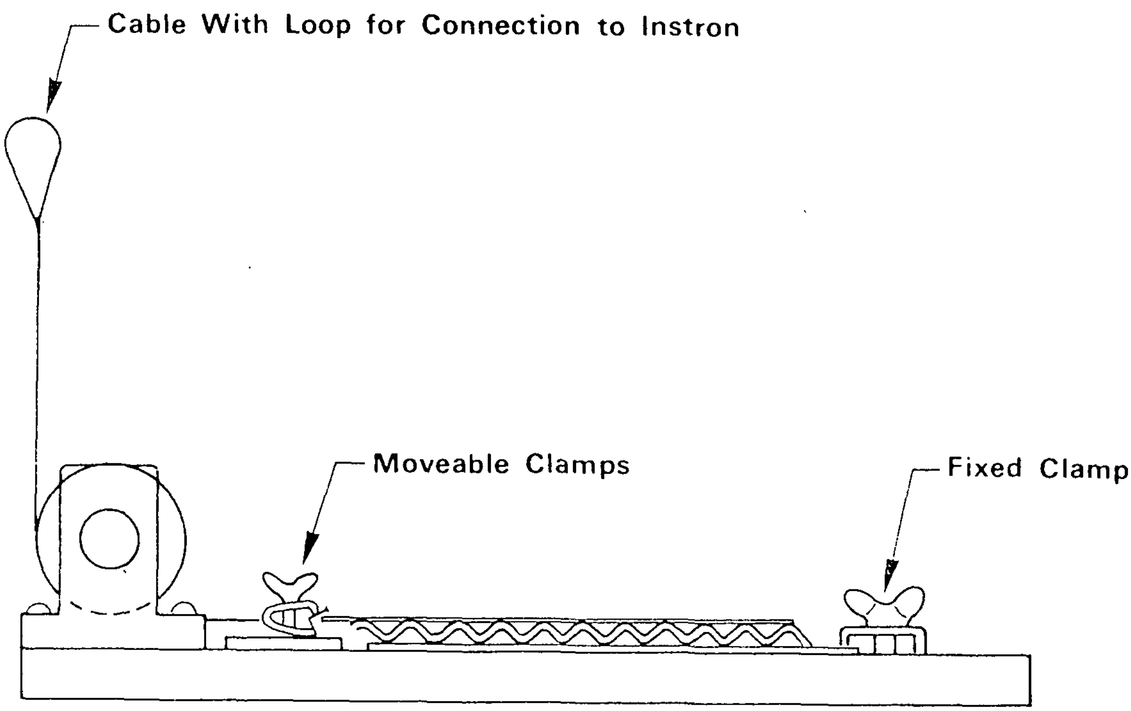


Figure 33. Wet shear strength test fixture.

b. Reference Tests on Conventional Water-Resistant Board

Several samples of regular production, hot corrugated, water-resistant board were tested in the wet shear test fixture to obtain representative numbers for comparison purposes. A 90-33-90 lb/MSF basis weight, C-flute, wet strength board and a 42-33-42 lb/MSF basis weight, C-flute, wet strength board were selected from the regular production of a member company. The heavy weight board failed in wet shear at an average value of 15.15 lb. The failures occurred in the single face glue line. The lighter weight board is the equivalent in basis weight to the combined board that was made via the cold process on the simulator. The wet shear test with that board failed at the single face glue line at an average load of 7.2 lb.

c. Development of a Cold Corrugating Water-Resistant Adhesive

Several commercial starches are available with higher than normal amounts of amylose. Conventional pearl corn starch, as used in the cold corrugating adhesive, has about 23% amylose. Some starch companies offer a special starch blend for use in formulating water-resistant hot corrugating adhesive with amylose contents in the range of 50-52%. Other formulations are available at even higher amylose content.

The initial trials on water-resistant cold corrugating adhesives were made with a 50-52% amylose starch called Hydro-Pruf, purchased from American Maize Products Company. Because this is a special blend for water resistance, other ingredients may be included but these were not identified. This starch was used in place of the conventional pearl corn starch with no other changes to the formula. When cooked at the standard 140°C temperature and 60 psi pressure, the Hydro-Pruff formula produced a water-resistant adhesive, but with an unstable viscosity. Adhesive made with the injection method of adding caustic was more stable than adhesive made with the post addition method of adding caustic, but neither one would be acceptable for production use. For reference purposes, the best wet shear strength value of the Hydro-Pruf adhesive was found to be 7.9 lb.

It had already been shown that cooking at higher temperatures provided increased stability for the amylose portion of the starch adhesives. When the Hydro-Pruf formulation was cooked at 160°C and 80 psi, the viscosity was nearly as stable as that of the nominal cold corrugating adhesive. However, no significant measure of water resistance could be found.

Another American Maize Products Company product called Amylomaize VII (AM VII) has about 70-75% amylose. When this starch was used in place of the pearl starch in the nominal formula and cooked at 160°C with the caustic injection method of pH control, a viscosity stable, water-resistant adhesive was produced. The wet shear strength value of this adhesive was found to be 8.4 lb. It should be noted that cooking AM VII at 140°C and/or post adding the caustic instead of injecting it in-line produced adhesive with unstable viscosities. Table XX summarizes all of the wet shear values discussed.

TABLE XX

WET SHEAR STRENGTH VALUES

Combined Board	Adhesive	Wet Shear, lb
90-33-90	Commercial hot corrugating	15.15
42-33-90	Commercial hot corrugating (as received)	7.2
42-33-90	Hydro-Pruf cold corrugating (unstable)	7.9
42-33-90	AM VII cold corrugating	8.4

Sufficient trials with the AM VII were conducted to determine that a threshold application rate exists somewhere between 0.5 and 1.0 lb/MSF. Below this level, poor water resistance was developed even though good combined board could be made on the simulator. Above this level, more adhesive did not appreciably increase the wet shear strength of the board. At an application rate of about 1 lb/MSF for each side, the AM VII adhesive would cost about 56¢/MSF (at the time these trials were conducted).

In some limited trials, addition of 1% itaconic acid to the AM VII slurry raised the wet shear failure load to 11-12 lb with some attendant fiber tear in the bond failure.

Some additional preliminary experiments on water resistant adhesive formulas have given some positive results by substituting a high amylose starch for the pearl starch used in a normal makeup and adding about 6% Amres. Amres, a reacting resin used in some conventional water resistant formulas, seems to further improve water resistance. Typically, combined board samples made on the simulator by using these formulas, cured at ambient conditions for 24 hours, and immersed in water for 24 hours show good bond integrity. Wet and dry burst values are shown in Table XXI.

TABLE XXI

WATER RESISTANT ADHESIVE PROPERTIES

	Basis Weights	Burst Values Dry, psi	Burst Values Wet, psi	% of Dry	Adhesive Application Rate
Cold corrugating	42/40/42	276	89	32	0.39 lb/MSF
Cold corrugating	42/40/42	261	88	34	1.08 lb/MSF
Hot corrugating	69/33/69	304	146	48	High but unknown

Application rates to achieve these results are as shown, so that adhesive cost for water resistant board would be below 50¢/MSF. Bond development curves suggest that water resistance board can be made at about the same speeds as for conventional products.

At this point in the development of water resistant adhesive, further effort was preempted by other urgent bonding issues. Unfortunately, those issues persisted throughout the remainder of the project, so no further work was done.

F. BONDING SYSTEM DEVELOPMENT DURING COMMERCIAL PROTOTYPE TRIALS 1981-1983

1. Introduction

The previous parts of this section of the report have described the early efforts to develop a cold corrugating adhesive, including the extensive attention to the double face bonding rate issue through use of the double backer simulator. Based on those studied, the pearl starch, setback adhesive was increased to maximum solids and converted at about 150°C for improved double face bond performance and adhesive stability. This adhesive, in conjunction with liner preheat and increased double backer combining pressure, provided the best overall double facing performance in both the pilot and commercial prototype trials. In accordance with the desire to use one adhesive for both bonds, this high solids adhesive was also used at the single facer in the commercial prototype trials.

After a lengthy process, the single face applicator system was finally successfully modified with a 10 quad gravure roll to provide a proper amount of adhesive. Single face bonds of adequate pin adhesion strength and CD/MD uniformity were then achieved. This applicator system and the high solids adhesive were used for the commercial prototype trials, discussed in Part III of Section IV. One of the three critical issues identified at the conclusion of those trials was toughness of the single face bond, a subjectively evaluated, but important property of combined board.

Figure 34 shows pin adhesion strength as a function of adhesive application rate. Although there is considerable scatter in these data indicated by the data band this relationship remained remarkably consistent through numerous changes in adhesive, applicator system and combining conditions. To achieve the required pin adhesion level of about 6.0 psi, it is necessary to apply about 1.4 lb/MSF of adhesive for the single face. This rate is high compared to that required for conventional corrugating (about 0.8 lb/MSF), but acceptable in terms of total adhesive cost and, probably, in terms of warp and washboarding. However, bond toughness, the deficient property, is not revealed by the pin value alone.

One typical measure of toughness is the locus of failure (LOF) criterion. Five failure zones are discernible: within the adhesive (AA), at the medium/adhesive interface (AC), in the medium (CC), at the liner/adhesive interface (AL), and within the liner (LL). For convenience, these are sometimes assigned integer numbers from 0 to 4, respectively. Ideally, the bond should fail in the liner (LL or 4) with failure at the other zones descreasing in desirability with the LOF number. Single face bond failure loci for the commercial prototype system are shown in Fig. 35 as a function of pin adhesion level and, through use of the relationship in Fig. 34, as a function of adhesive application rate. The toughness problem is clearly evident from this data presentation. To guarantee bond toughness, i.e., bond failure in the liner, the

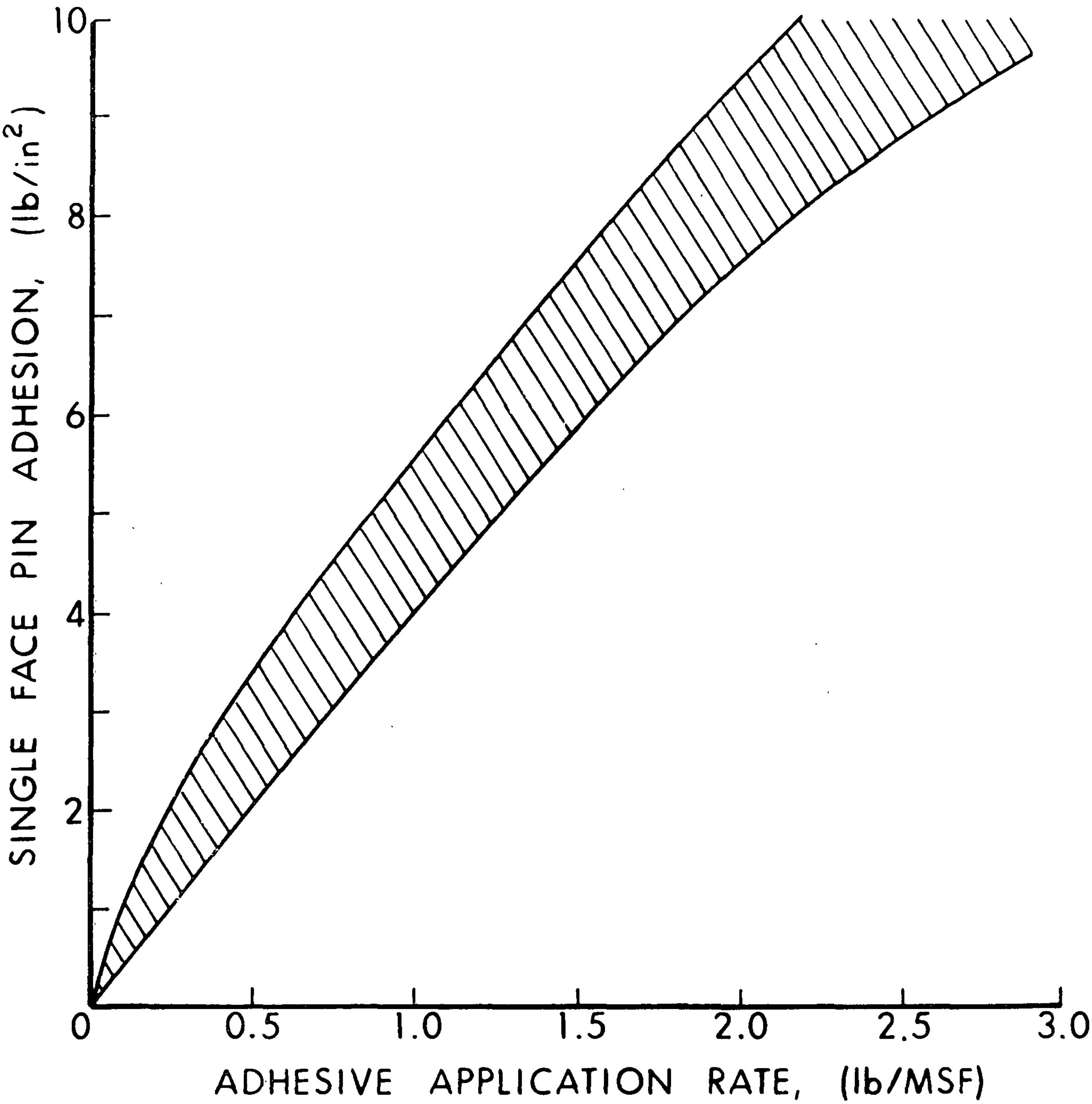


Figure 34. Single face pin adhesions *versus* adhesive application rate.

adhesive application rate must exceed about 2.5 lb/MSF. The corresonding pin adhesion level is about 10 psi, which is higher than actually needed for strength. The application rate required to get toughness is regarded as too high, however,

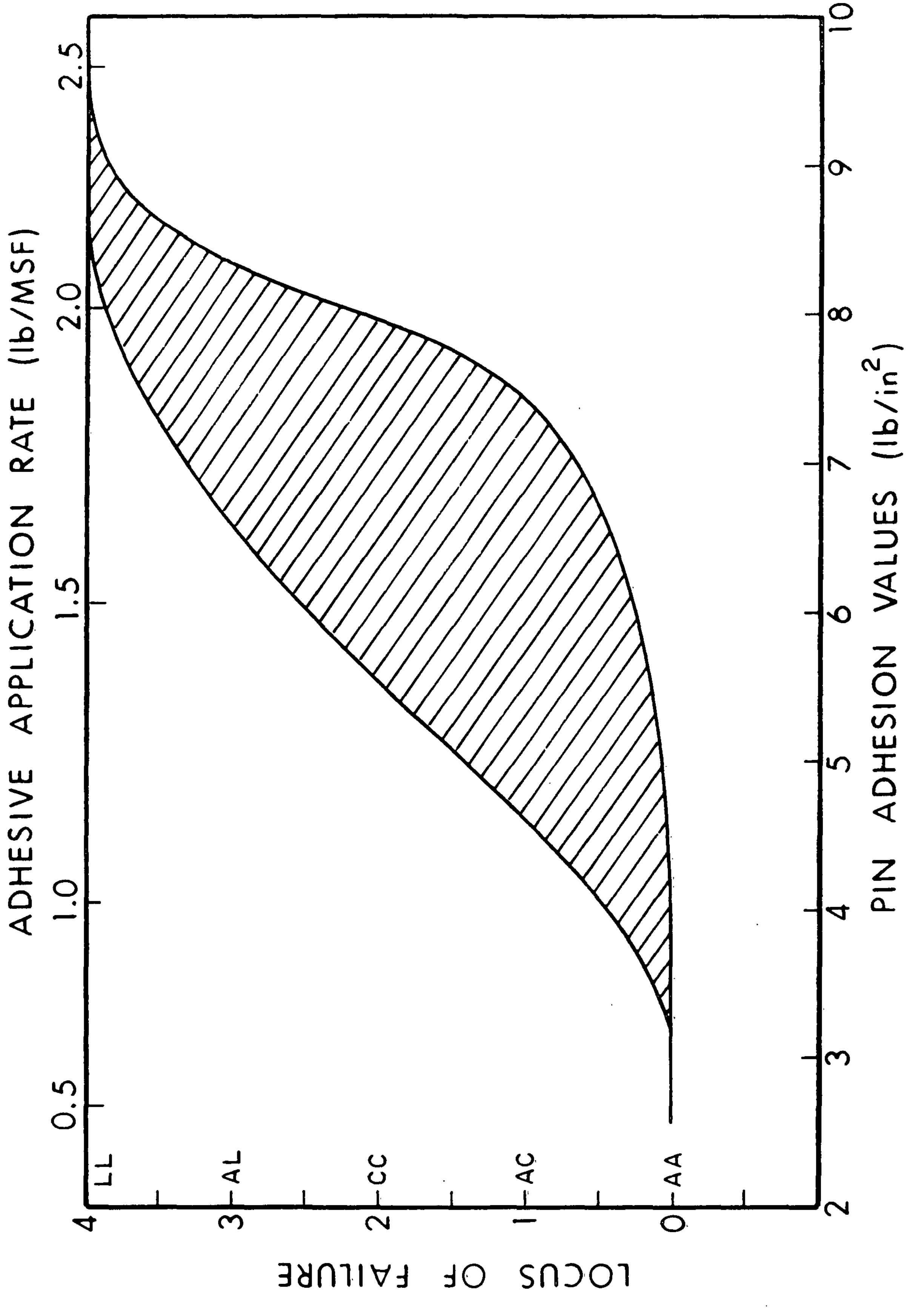


Figure 35. Relationship of pin adhesion values and locus of failure.

because of the corresponding high cost, warp, washboarding and water added. The basic single face bond toughness issue, finally identified correctly in the commercial prototype trials, is that of achieving both an adequate pin adhesion level and bond failure within the liner at an attractive adhesive application rate, preferably below 1.25 lb/MSF. During 1983, this problem was vigorously pursued in the laboratory. Before describing this work some additional historical perspective on the issue is given below.

2. Background on Single Face Bonding

The early development work on "cold" corrugating adhesives, described in some detail earlier in this part of the report, was directed to developing adhesive for a "warm" corrugator. In this early approach, the corrugating rolls remained hot and the pressure roll was operated cold. Most adhesive formulas of that era were low in solids (20-25%) compared to later versions and heavily chemically converted because of the limited thermal conversion capability of the short dwell time (6 sec) cooker used at that time. Bond brittleness, or lack of toughness, was recognized as an issue at that time.

As the project progressed, solids levels increased, the bisulfite was dropped from the formula and, eventually, in the cold corrugating feasibility trials, the heat was removed from the corrugator. Generally, as these steps were taken, toughness improved, but remained as an issue.

About 1977, the project moved to demonstration of technical feasibility as an objective and to the building of pilot scale starch conversion equipment. With this equipment, thermal conversion of the starch was increased, chemical conversion reduced, and the dwell time increased sufficiently to exhaust the conversion chemicals. Control of adhesive application rate remained a problem, a severe one, on the pilot equipment. Under these conditions single face bonds were generally strong and tough enough to satisfy commercial demands. Again, the true impact of excessive adhesive application rate was not fully understood. Furthermore, the initial pilot trials revealed the true severity of the bonding challenge at the double facer. For these reasons, the bonding system development work was largely deflected to the double facer issue until the single face bond deficiency reemerged as a critical problem in the commercial prototype trials.

A confounding issue in all of the early work was the absence of measurement and control of adhesive application rate. Because of the higher solids levels and limited shear thinning of the setback adhesive, the actual application rate in pounds of starch per MSF was usually higher (based on subsequent data) than believed at the time. As is now known, a high application rate tends to mask the brittleness problem, so the originally recognized deficiency was probably greater than realized. Hence, it did not receive as much attention in development work and decision as, perhaps, it would have had better quantification tools been available.

3. Laboratory Investigation of Single Face Bond Toughness

Once recognized as perhaps the critical issue for the ultimate success of cold corrugating, bond toughness was pursued by detailed investigations of three of the four factors in the bond system: adhesive chemistry and rheology, including additives; adhesive application, including amount and distribution; and combining conditions at the bonding nip (pressure roll-lower corrugating roll). The basic objective was to achieve adequate strength and toughness with about 1.25 lb/MSF or less of adhesive.

a. Assessment Techniques

The locus of failure in a pin adhesion test has been traditionally used as an indicator of bond toughness in laboratory analyses. While effective, this test lacks the resolution desired for quantitative assessment in laboratory work. In the field, toughness is usually subjectively evaluated by peeling the V-shaped pieces of liner resulting from an X-cut in the liner face. A sharp pull in the peeling direction on the tip of the V will split the liner if the bond is tough; for brittle bonds, failure will occur in the adhesive film between components. This test is even less quantitative than the LOF test.

In an attempt to provide a repeatable, quantitative toughness test, combined board strips 1 inch wide by 20 inch long were cut. The liner was then "peeled" from these samples in an Instron tester to measure the force-time history for failure of a typical bond. Various rates of loading were examined in an attempt to identify an effective test procedure. Typical load-time recordings for a sample of hot corrugated board, known to possess tough bonds, and for a representative cold sample, are shown in Fig. 36A and B. These curves are clearly different in several respects. Attempts were made to use the area under the curve (energy to failure), the curve shape (one peak or two), and the maximum peeling load as toughness indicators. Energy to failure (area under the curve) was sensitive to toughness and correlated with LOF as well as the inherent scatter in such data would allow. However, upon completion of this investigation, it was concluded that the much more costly and tedious Instron test was no better than the LOF. Consequently, the LOF test was adopted for use in the remainder of the study.

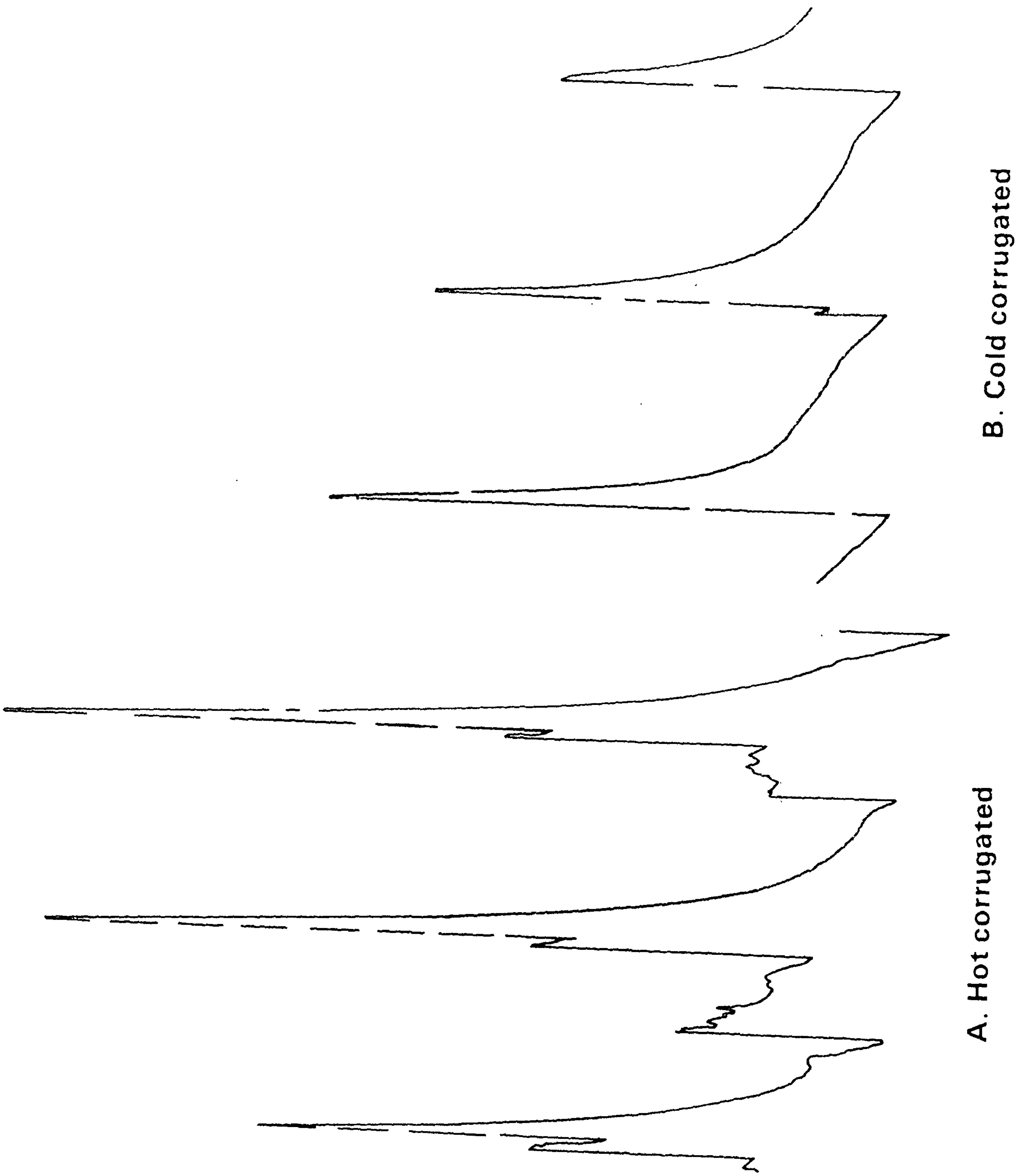


Figure 36. Load-time recordings from "peel" test.

In this investigation extensive use was made of photomicrographs of bond cross sections. A typical example is shown in Fig. 37. While only "snapshots" at one spot on the bond line and, thus, a potentially poor representative of the average bond, the micrographs were very useful in determining the general character of the bond. Such features as adhesive distribution through and around the bond zone and the nature of the contact between the flute tip and the liner were readily discernible.

For purposes of this study, a generally complete assessment of a single face bond included the following elements;

1. A pin adhesion test for bond strength
2. A locus of failure test for toughness
3. Application rate for cost, warp, etc.
4. A photomicrograph for visual assessment
5. Combining conditions for an assessment of commercial practicability

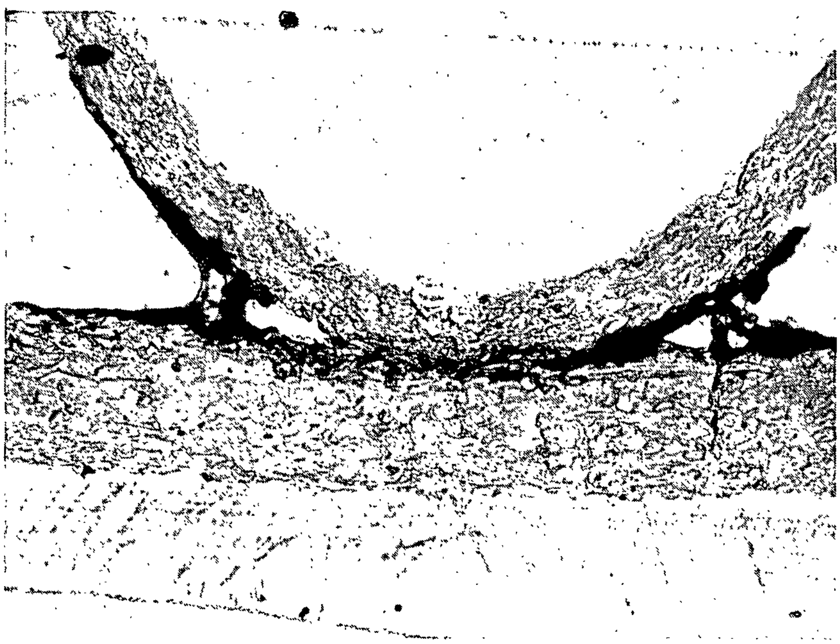


Figure 37. Typical photomicrograph of cold corrugated single face bond.

6. Taper grinding and iodine staining to indicate geometric distribution of starch.

None of these evaluation techniques has enough resolution or precision to find small changes (10-15%). However, changes of the magnitude desired (> 50%) should be readily detectable and, perhaps, quantifiable with these procedures.

b. Physical Analysis of Single Face Bonds

The strength and locus of failure data in Fig. 34 and 35 show the gross failure properties of single face bonds but offer little help in understanding bond brittleness. A photomicrograph of a representative single face bond taken from one of about 10,000 boxes produced to fill a commercial order is shown in Fig. 37. For comparison, a similar photograph for a hot corrugating bond is shown in Fig. 38. Several important physical features, discernible in Fig. 37, appear important to bond toughness. These include:

1. The bonded area is fairly narrow - the equivalent of about 4-1/2 times the medium thickness. For hot corrugating, the bond width is about 25-30% greater.
2. The fillet region of the bond is largely devoid of adhesive. This reduces the bonded area and probably contributes to a stress concentration at the bond edge under load (filleted or rounded boundaries tend to distribute applied loads and reduce local stress levels).
3. While there is a "bridge" of adhesive between the medium and liner outboard of the primary bond zone, it appears ineffectual in contributing to bond strength. This is confirmed by physical testing.

Figure 38. Typical photomicrograph of hot corrugated single face bond.

4. The primary compression zone (from loading in the bonding nip) appears to extend to the adhesive bridge area. However, the completed bond shows only limited permanent (retained) compressive deformation of the liner, much less than that for the hot corrugating bond. Separate tests show that the maximum compressive deformation is much greater, so significant "rebound" must occur as the joint leaves the bonding nip.

5. A large amount of adhesive appears on both the medium and the liner well outboard of the bond zone. Simple geometric analysis of the joint under maximum compressive

deformation shows that the adhesive is not squeezed into these regions. Apparently, then, the adhesive is ejected from the joint by the high pressure impulse applied at the bonding nip. Surface tension forces are probably responsible for splitting the film to give the nearly equal distribution between the medium and liner.

Taper grinds of the bond joint, followed by iodine staining of the starch, show that penetration of both the medium and liner is great, usually 4 mils or more and sometimes 8 mils. The relative narrowness of the cold single face bond is also confirmed by these tests.

The failure of a tough single face bond in a pin adhesion test is distributed over time with several partial failures and recoveries, each occurring as a liner fiber tear initiates and then stops. A final failure time is usually not sharply defined. This type of failure, evident from the multiple secondary peaks in the peeling load trace in Fig. 36A, produces a very distinctive tearing sound.

Failure of a brittle single face bond is very different. In a pin test there may be some popping and cracking from the sample as it is loaded, but partial failures and recoveries do not occur. At the maximum load, failure occurs suddenly and catastrophically, accompanied by a load popping sound. All or a large part of the liner may separate cleanly and at one time. This type of failure is illustrated by the peeling load trace in Fig. 36B which shows a smooth rise to one peak, followed by a complete loss of load carrying ability.

For comparison, a photomicrograph of a typical double backer simulator bond is shown in Fig. 39. This bond was made with an adhesive application rate of about 0.5 lb/MSF. The pin adhesion value was about 7.5 psi and failure occurred in the liner. Hence, this is generally a good quality bond. Note that the bonded width is about the same as for the cold single face sample. Liner penetration for such samples is typically 2-3 mils, considerably less than for the single face bond. Almost all of the applied adhesive is effective in producing bond strength.

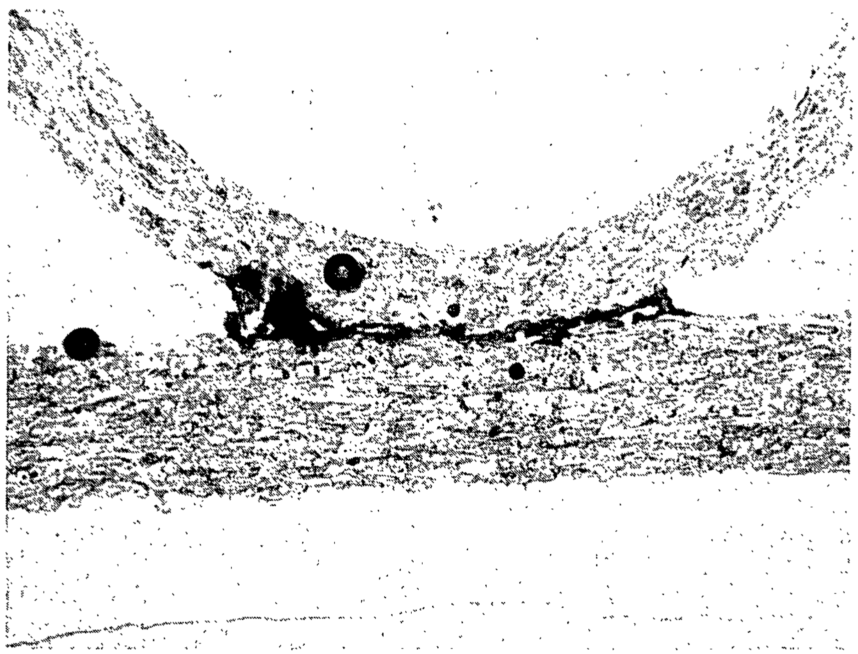


Figure 39. Photomicrograph of double backer simulator bond.

One useful measure of bonding system effectiveness is the total carrying potential produced by one pound of adhesive. Some estimated effectiveness numbers are shown below:

Bond Type	Effectiveness lb_f/lb_m
Cold	
Double backer simulator	3,000,000
Single face	700,000
Double face	1,000,000
Hot	
Single face	1,400,000
Double face	700,000

These data clearly show that neither of the processes is effective in capturing the inherent strength of a starch adhesive.

c. The Single Face Bonding Sequence

To produce a single face bond, adhesive is applied to the tips of the flutes with the amount and distribution controlled by the applicator systems. After a short open time, the medium and liner are brought together in the bonding nip with the adhesive sandwiched between them. For cold corrugating, it appears that the adhesive does not wet the medium nor cool significantly during the open time, especially at high speeds. Thus, the adhesive is quite mobile when it arrives at the bonding nip. As the sandwich passes through this nip it is subjected to a short, high pressure pulse or impulse. Mechanical dewatering and adhesive penetration into the components, both driven by this pressure impulse, cause the formation of a green bond. This process occurs in just a few milliseconds. As the sandwich leaves the nip the compression forces are relieved and the medium rebounds partially from the liner. As a result, part of the initial contact zone opens completely. The excess adhesive, originally ejected from the contact zone, does not flow back to fill the voids, so the bonding potential of this area is lost. After the single face board leaves the nip a final bond forms slowly (in a few seconds) as the remaining water is removed through naturally occurring transport processes.

Bond strength and toughness seem to be limited by the narrow bond zone and by stress concentration at the bond edges. Low cohesive strength may be a factor, but the quality of the double backer simulator bonds suggests otherwise. Effectiveness of the bonding system appears limited by adhesive ejection (the ejected adhesive does not contribute to bond performance), by excess penetration, and by springback of the medium and liner. The higher mobility of the adhesive at the bonding nip allows ejection to take place.

In the foregoing paragraphs, we have described the bond toughness problem, presented the physical characteristics of the bond which may be related to toughness, and described the sequence of events in the formation of a bond. Based on these data, five possible avenues for improving bond toughness were identified and explored: adhesive cohesive strength, adhesive rheology, surface interfacial phenomena, adhesive application, and combining conditions. For each of these the rationale, experiments and results are described briefly below.

d. Adhesive Formulation

Several adhesive formulation changes were evaluated for their affect on bond toughness through corresponding changes in cohesive strength, rheology, or medium wetting. Even for the adverse physical structure of the single face bond shown in Fig. 37 increases in adhesive cohesive strength should give increases in bond toughness. Adhesive rheological and wetting properties determine the migration of the adhesive in response to the driving forces imposed from application through combining. Changing these properties to improve wetting and resist ejection should improve bond toughness. Each adhesive formulation change was evaluated in hopes of influencing one or more of these factors. Assessments were carried out by measuring bond properties rather than by direct evaluation of adhesive properties.

Table XXII contains a list of all the formulation changes that were investigated along with a brief rationale for doing so and key results from these experiments.

None of the adhesive formulation changes produced significant positive results on a consistent basis.

TABLE XXII

ADHESIVE FORMULATIONS

Formula in Change	Range	Rational	Expected to Affect: Strength/ flexibility	Expected to Affect: Rheology	Expected to Affect: Wetting
Amylose/amylo-pectin ratio	0-100%	Change properties of straight and branched chains	X	X	
Thermochemical condensation ratio	280-320°	Change polymer size distribution	X	X	
Wetting agents		Improve wetting			X
Viscosity	150-900 Brabender	Change polymer size distribution	X	X	
Add wood fiber	3%	Change rheology and reinforce film	X	X	
Vary boric acid content	0-0.4%	Change tack and rheology	X	X	
Lower starch solids	32%	Change rheology		X	
Add clay filler	35%	Change rheology		X	

e. Adhesive Application

From Fig. 37, the final adhesive distribution in and around the bond zone is far from optimum, with much of the adhesive contributing little to bond performance. This final distribution is the net result of the distribution as applied, modified by the migration induced by the pressure impulse at the bonding nip. Fluid flow induced through naturally occurring gradients is believed to be negligible under the prevailing circumstances.

From this analysis, initial placement of the adhesive on the flute tip may partially determine the final distribution. Intuitively, concentrating the adhesive in a thin line at the center of the tip should help to "trap" the adhesive and prevent ejection. Unfortunately, the flute tip geometry is ill-suited to this process, but well-suited to enhance the ejection process. Various schemes for altering the geometry to a more favorable form by flattening, dimpling or roughening the flute tip were considered, but rejected as unworkable in a practical corrugator.

To test the importance of initial adhesive placement, the pilot single facer was adjusted to establish minimal contact between the flute tip and the adhesive film. This proved extremely difficult to do because of medium fluff-out on this fingered machine and the relatively poor mechanical condition of the machine. Nevertheless, some tests were conducted with very thin lines of adhesive deposited at the flute center.

When this form of application was used with normal pressure roll conditions the results were poor. Data from these experiments, along with many others, show that the final adhesive distribution and, hence, bond toughnes, is dominated by the pressure roll ejection process and not by initial placement. A typical photomicrograph is shown in Fig. 40. These data largely explain the disappointing single face bonding results obtained with gravure applicator rolls. These were expected to apply the adhesive uniformly over the flute tip, which in turn was expected to improve dewatering for green bonding and effectiveness in the final bond.

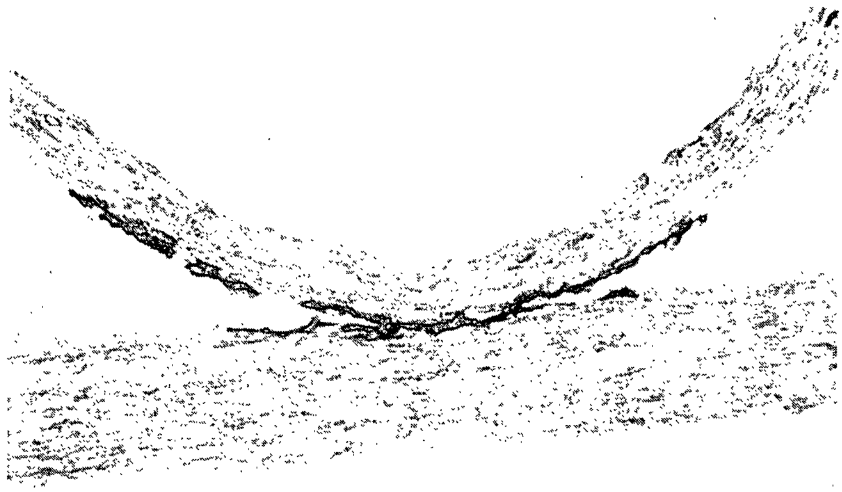


Figure 40. Photomicrograph of cold corrugating single face bond with low adhesive application rates.

The overwhelming dominance of the ejection forces also explains the lack of positive results from adhesive rheological changes. Massive changes in rheology would be required to counter the ejection forces. Such large changes would not be workable in any practical system.

Despite the general insignificance of initial adhesive distribution to bond toughness and effectiveness, symmetry remains an important issue. Several trials were conducted at different ratios of applicator roll surface speed to machine speed (A/C) to test the importance of altered symmetry in the adhesive pattern. Unfortunately, varying A/C changes both symmetry and the application rate, making it difficult to isolate the symmetry effect. For the laboratory single facer, the data seem to slightly favor A/C = 0.95. With this ratio, the flute tips wipe forward slightly to place more adhesive on the leading edge of the flute tip. This tendency is partially compensated by backward redistribution of the adhesive in the film splitting process. Although the new applicator system for the Savannah single facer was designed with A/C = 0.95, this is not believed to be an important factor in bonding performance.

f. Combining Conditions

The investigations described above showed that changes in adhesive chemistry and application were largely ineffective in improving bond toughness, at least within the ranges allowed by practical application. These same investigations revealed the extreme severity of the ejection process and its insensitivity to adhesive rheology. As a consequence, increased attention was given to the single facer combining conditions already recognized as important.

Two conflicting processes take place at the bonding nip: dewatering of the adhesive to form a green bond, which is desirable and requires high pressures, and adhesive ejection which is very undesirable and increases with pressure. Successful single face bonding requires proper consideration of both.

From pressing theory, it is known that mechanically induced dewatering is often directly proportional to impulse, where impulse is the area under the pressure-time curve. Dewatering under wet conditions (green bonding) is especially likely to be impulse controlled. In this dewatering regime (impulse controlled) pressure and time may be freely interchanged without affecting dewatering, as long as the total impulse remains constant. Hence, for green bonding, a long nip residence time coupled with a low nip pressure should be as good as conventional pressure roll nips with high pressures and short times. A low nip pressure should produce less adhesive ejection and, hence, better bond toughness. Nip residence time can be increased by running at low speeds (which is useful for testing but not for production) or by softening the pressure surface to increase the effective nip width.

Table XXIII presents summary data from a few of many experiments conducted to test low pressure combining with various pressure roll covers. Micrographs from a few of the best experiments, shown in Fig. 41, are labeled to correspond to the table.

TABLE XXIII

DATA FROM LOW PRESSURE COMBINING CONDITIONS

Cover	Stops[a]	Load, pli	Pin Adhesion Test, lb/in^2	Pin Adhesion Test, LOF	Figure	Speed, fpm
90 lb liner	0	300	77	AA		20
90 lb liner	0.010	300	98	LL		20
None	0	300	98	AA	42A	100
None	0.012	300	111	LL	42B	100
None	0.018	200	40	AL	42C	20

[a]0-indicates stops adjusted so pressure roll is just making contact with the lower corrugating roll, 0.010 indicates a 0.010 inch gap.

A micrograph from one of the most interesting special combining experiments is shown in Fig. 42. The special adhesive applicator system adjustments, described earlier, were used to apply the adhesive in a thin line along the center of the flute tip. By special procedures the pressure roll gap was set at about 0.016 inch to limit compression (pressure) in the nip. The machine was operated at low speeds to provide maximum nip residence time (impulse under low pressure conditions). Performance data, summarized in Table XXIV, show the excellent results obtained. These data are supported by the micrograph.

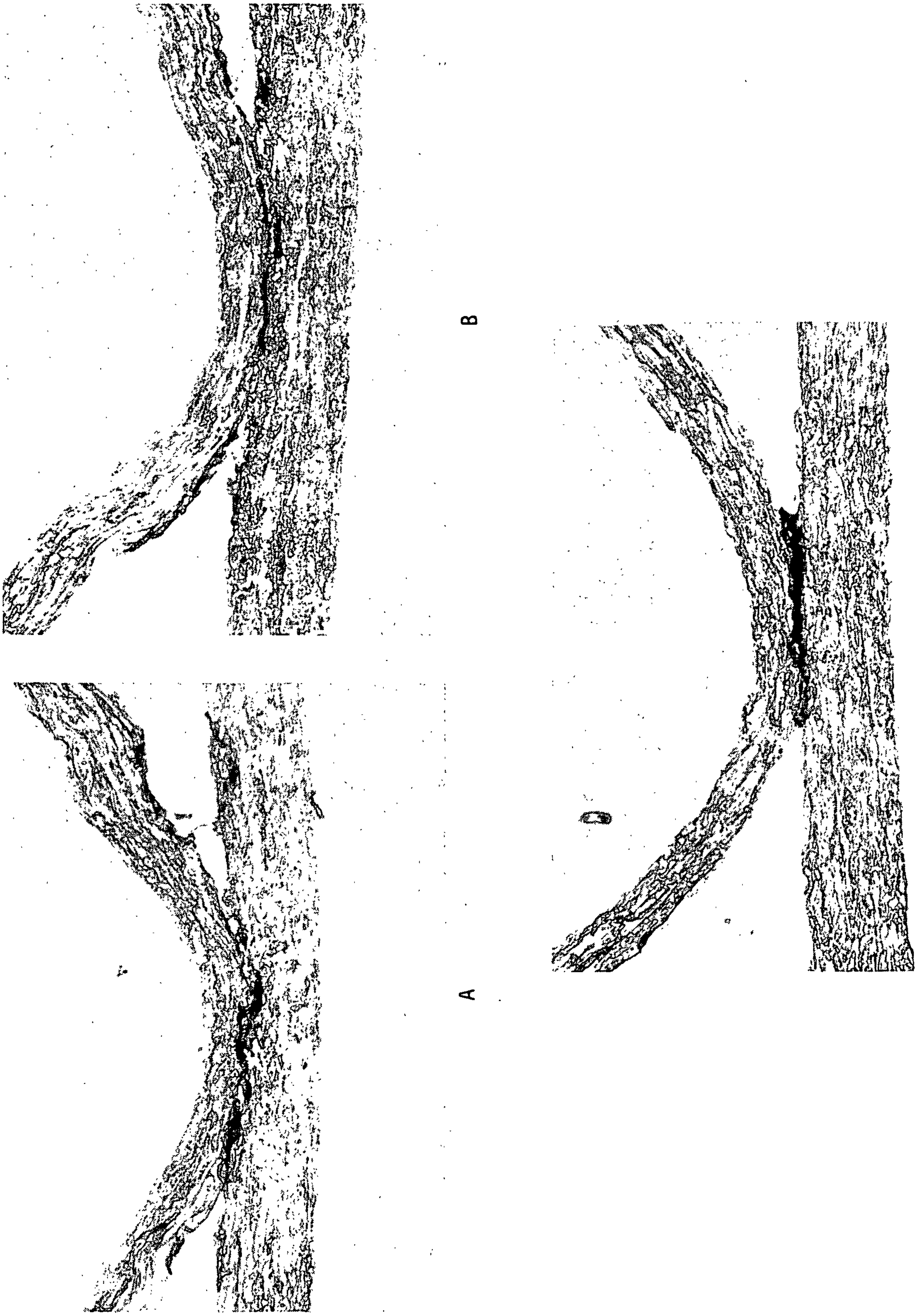


Figure 41. Photomicrographs of cold single face bonds formed with various combining conditions.

TABLE XXIV

PERFORMANCE FACTORS

Adhesive application rate	0.70 lb/MSF
Pin adhesion value	7.6 psi
Locus of failure	LL
Bond effectiveness	1,600,000

Despite the low adhesive application rate, the pin adhesion levels are high and failure occurred within the liner. The bond effectiveness value is nearly twice that for normal single facer operation. From the micrograph there was no adhesive ejection, so all the adhesive was effective in contributing to the bond. The green bonds, subjectively evaluated at the exit of the nip, were excellent, producing fiber pull at this very early stage.

At higher speeds (> 100-200 fpm) the available impulse was too low to produce a satisfactory green bonding, thus precluding faster machine operation. These data are typical of the results from a number of experiments conducted to test the basic idea of a low pressure, extended nip. The concept appears to be valid and workable, but practical implementation within the constraint of minor modification to a normal machine design seems unlikely.

In one limited test of the idea, a rubber covered roll was installed in the single facer in Savannah. The roll cover was 1/4 inch thick with an equivalent hardness of 4 P&J. Only very limited trials were conducted, but the results were not very encouraging. However, to realistically test a wider nip concept, it would be necessary to fully analyze and redesign the roll stack for the lower nip pressures required. This was not and could not be done. Furthermore, at the time these trials were being conducted, the project was concluded by mutual agreement of the participating parties. Hence, the idea was not properly implemented, nor given a fair trial. Nevertheless, the implementation process appears difficult.

Figure 42. Photomicrograph of cold single face bond.

4. Summary

The only truly effective mechanism for improving bond toughness emerging from this work is low pressure, extended time combining. This appears to offer significant benefits in bond performance, including green bonding, final bond strength, toughness, and effectiveness. Similar benefits might acrue to the hot process as well, since the bonding mechanisms at the single facer are somewhat similar. Two approaches seem open to solving the problem: change the combining system to provide adequate dewatering but without adhesive ejection or completely change the adhesive concept. Neither was regarded as feasibile, given the magnitude of the task and the status of the project. Furthermore, both are best carried out in a laboratory setting, not on a commercial prototype

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